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The Journal *of the* Society of Dyers and Colourists

Volume 74 Number 2

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THE SOCIETY OF DYERS AND COLOURISTS
DEAN HOUSE PICCADILLY BRADFORD YORKSHIRE

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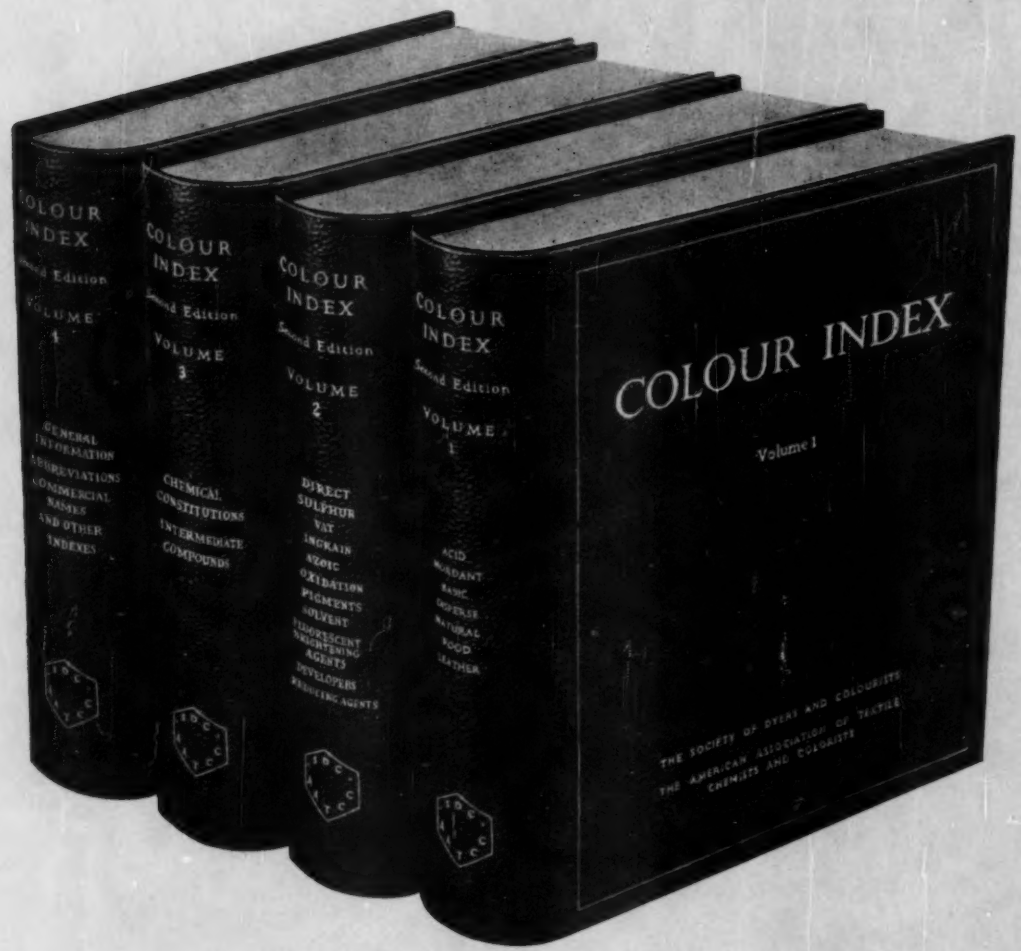
and

THE AMERICAN ASSOCIATION OF TEXTILE CHEMISTS AND COLORISTS



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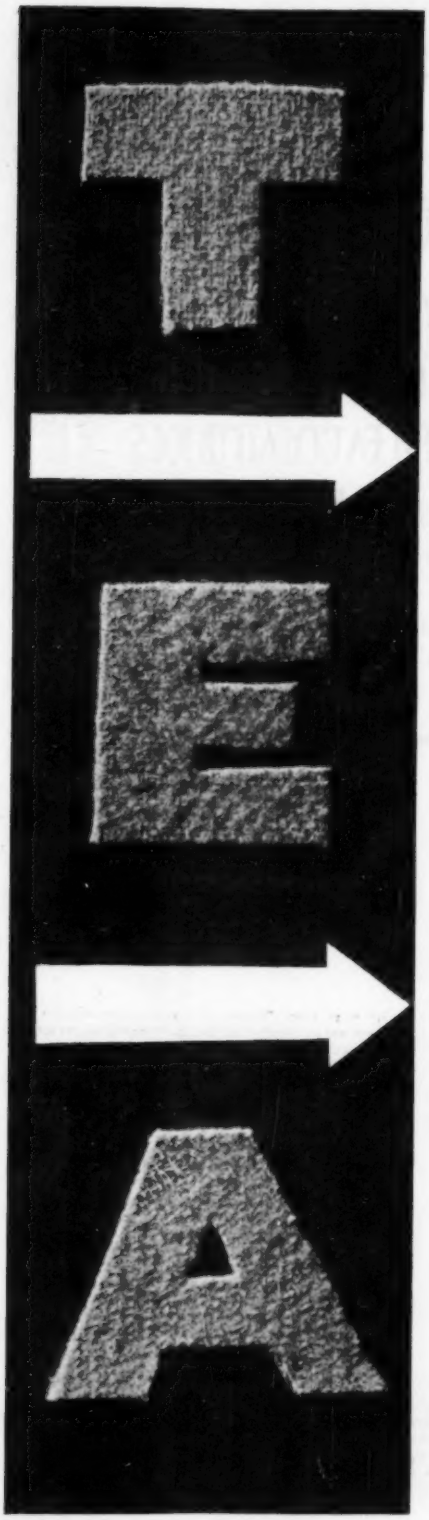

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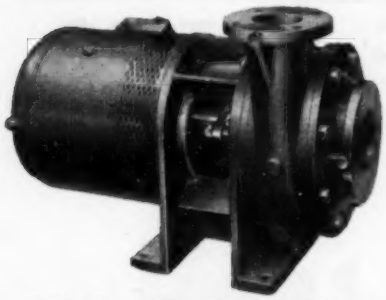

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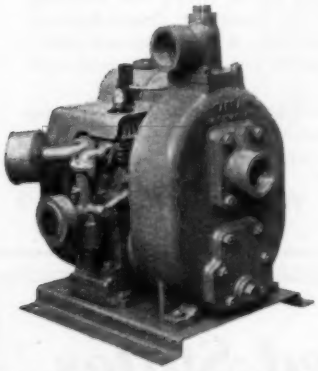

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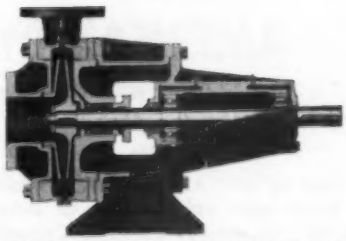

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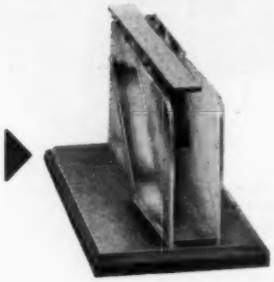

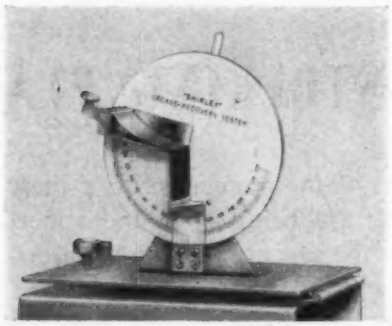

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–7 of the January 1958 and pages 285–290 of the July 1957 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

COMMUNICATIONS

A Dye-uptake Meter for Studies of Dyeing Kinetics — *E. Bakker and D. Patterson*

A Study of the Physical State of Methylene Blue in Dyed Films and its Relation to Light-fading Rates — *D. S. E. Campbell and C. H. Giles*

Studies in Aminoanthraquinone Compounds
I— Absorption Spectra in Solution and in the Solid State — *G. S. Egerton and A. G. Roach*

Effect of Sulphuric Acid on Wool
I— Histological Attack at Various Concentrations
II— Chemical Attack of Concentrated Acid
R. L. Elliott, R. S. Asquith, and D. H. Rawson

The "Pad–Jig" System of applying Vat Dyes to Linen Piece Goods — *J. N. F. Phillips*

The Determination and Significance of Crystallite Size in Regenerated Cellulose Fibres — *D. N. Tyler and N. S. Wooding*

EXPLANATORY PAPER ON MODERN THEORY

Surface Activity, Solution, and Adsorption — *C. H. Giles*

JOURNALS WANTED

The Society is urgently wanting Journals (in good condition) for all months of 1955 except July and December. The months of January, February, March, August and September 1955 are particularly required. Copies of January, February, March, April, July, August, September, October and December 1956 issues are also in demand.
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FORTHCOMING MEETINGS OF THE SOCIETY

Tuesday, 25th February 1958

LEEDS JUNIOR BRANCH. *Problems in the Dyeing of Wool, Fibro and Terylene for Blends.* Miss E. Smith., The Departmental Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, **Leeds 2.** 3.15 p.m.

Thursday, 27th February 1958

WEST RIDING SECTION. *The Reactivity of Keratin.* (Joint Meeting with the Yorkshire Section of The Textile Institute.) Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I. The Victoria Hotel, Bridge Street, **Bradford.** 7.30 p.m.

Tuesday, 4th March 1958

LEEDS JUNIOR BRANCH. *Optical Bleaching Agents.* D. A. W. Adams, Esq., Ph.D. Department of Colour Chemistry and Dyeing, The University, **Leeds 2.** 3.15 p.m.

Thursday, 6th March 1958

SCOTTISH SECTION. *Continuous Dyeing of Wool.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Scottish Woollen Technical College, **Galashiels.** 7.15 p.m.

Friday, 7th March 1958

BRADFORD JUNIOR BRANCH. *The Dyeing of Cellulosic Fibres with Alcian X Dyestuffs.* J. T. Turner, Esq. (I.C.I. Ltd.). Bradford Institute of Technology, **Bradford.** 7.15 p.m.

LONDON SECTION. *Recent Developments in the Dyeing of Man-made Fibres.* Dr. R. Wittwer (Ciba Ltd.). Royal Society, Burlington House, **London, W.1.** 6 p.m.

Tuesday, 11th March 1958

HUDDERSFIELD SECTION. *Recent Developments in the Dyeing of Man-made Fibres.* To be read by a member of The Clayton Dyestuffs Co. Ltd. Silvios Cafe, **Westgate, Huddersfield.** 7.30 p.m.

NORTHERN IRELAND SECTION. *Making the Best Use of Levelling Agents.* G. H. Lister, Esq., B.Sc., Ph.D. (Sandoz Products Ltd.). Thompson's Restaurant, **Belfast.** 7.30 p.m.

Wednesday, 12th March 1958

SCOTTISH JUNIOR BRANCH. Annual General Meeting. 7 p.m.
Lecture 7.45 p.m. *The Drying of Textiles.* K. S. Laurie, Esq., M.A., A.M.I.Mech.E., A.M.I.E.E. (John Dalglish & Sons Ltd.). Technical College, **Paisley.**

Thursday, 13th March 1958

MANCHESTER JUNIOR BRANCH. *Terylene.* J. R. Whinfield, Esq. Room C9, Manchester College of Technology, **Manchester.** 4.30 p.m.

WEST RIDING SECTION. Discussion, *Fluorescent Brightening Agents.*

Panel
- A Member of the British Cotton Industries Research Association.
- J. S. Ingham, Esq. (Marks & Spencer Ltd.).
- Dr. Fearnley (Geigy Co. Ltd., Manchester).
- J. Rayment, Esq. (G. & W. N. Hicking).

Victoria Hotel, **Bradford.** 7.30 p.m.

Tuesday, 18th March 1958

SCOTTISH SECTION. *Easy Care Finishes on Fabrics Containing Courpleta, with special reference to Pleating, Embossing and Setting.* A. S. Cluley, Esq., F.T.I. and S. N. Bradshaw, Esq. (Courtaulds Ltd., Coventry). St. Enoch Hotel, **Glasgow.** 7.30 p.m. To be preceded by Sectional Annual General Meeting at 7 p.m.

Wednesday, 19th March 1958

MIDLANDS SECTION. *Optical Brightening Agents.* D. A. W. Adams, Esq., B.Sc., Ph.D. (Hickson & Welch Ltd.). (Joint meeting with The British Association of Chemists.) Midland Hotel, **Derby.** 7 p.m.
Historical, present market position, mode of action and general properties, special characteristics for specific uses, chemical constitution of main types, evaluation.

Friday, 21st March 1958

MANCHESTER SECTION. One-day Symposium. *New Information on Finishing Processes.* Manchester College of Science and Technology, **Manchester.**

MIDLANDS SECTION. Section Annual Dinner. George Hotel, **Nottingham.**

Saturday, 22nd March 1958

NORTHERN IRELAND SECTION. Annual General Meeting and Dinner. Thompson's Restaurant, **Belfast.** A.G.M. 5 p.m, Dinner 6.30 for 7 p.m.

Tuesday, 25th March 1958

HUDDERSFIELD SECTION. Annual General Meeting.

Thursday, 27th March 1958

BRADFORD JUNIOR BRANCH. *Continuous Dyeing of Wool.* D. R. Lemin, Esq., B.Sc. (I.C.I. Ltd.). Bradford Institute of Technology, **Bradford.** 7.15 p.m.

WEST RIDING SECTION. Annual General Meeting. Victoria Hotel, **Bradford.** 7.30 p.m.

Saturday, 29th March 1958

BRADFORD JUNIOR BRANCH. Annual General Meeting. Bradford Institute of Technology, **Bradford.** 10.15 a.m.

Thursday, 3rd April 1958

MIDLANDS SECTION. *Recent Trends in Dyeing for the Carpet and Hosiery Trades.* W. Beal, Esq., B.Sc. (Geigy Co. Ltd.). Messrs. Carpet Trades Ltd. Canteen, Mill Street, **Kidderminster.** 7.30 p.m.
(Joint with the Textile Institute, Kidderminster Section.)

Wednesday, 16th April 1958

MIDLANDS SECTION. *Basic Principles of Dyeing with Disperse Dyes.* D. Finlayson, Esq., M.C., M.A., D.Sc. and H. C. Olpin, Esq., M.Sc., F.R.I.C., F.S.D.C. King's Head Hotel, **Loughborough.** 7 p.m. (Preceded at 6.30 p.m. by the Annual General Meeting of the Section.)

Friday, 18th April 1958

LONDON SECTION. *Colour Fastness Requirements of Luton Textiles.* I. Glasman, Esq., A.T.I. (Marks & Spencer Ltd.). George Hotel, **Luton.** 7 p.m.

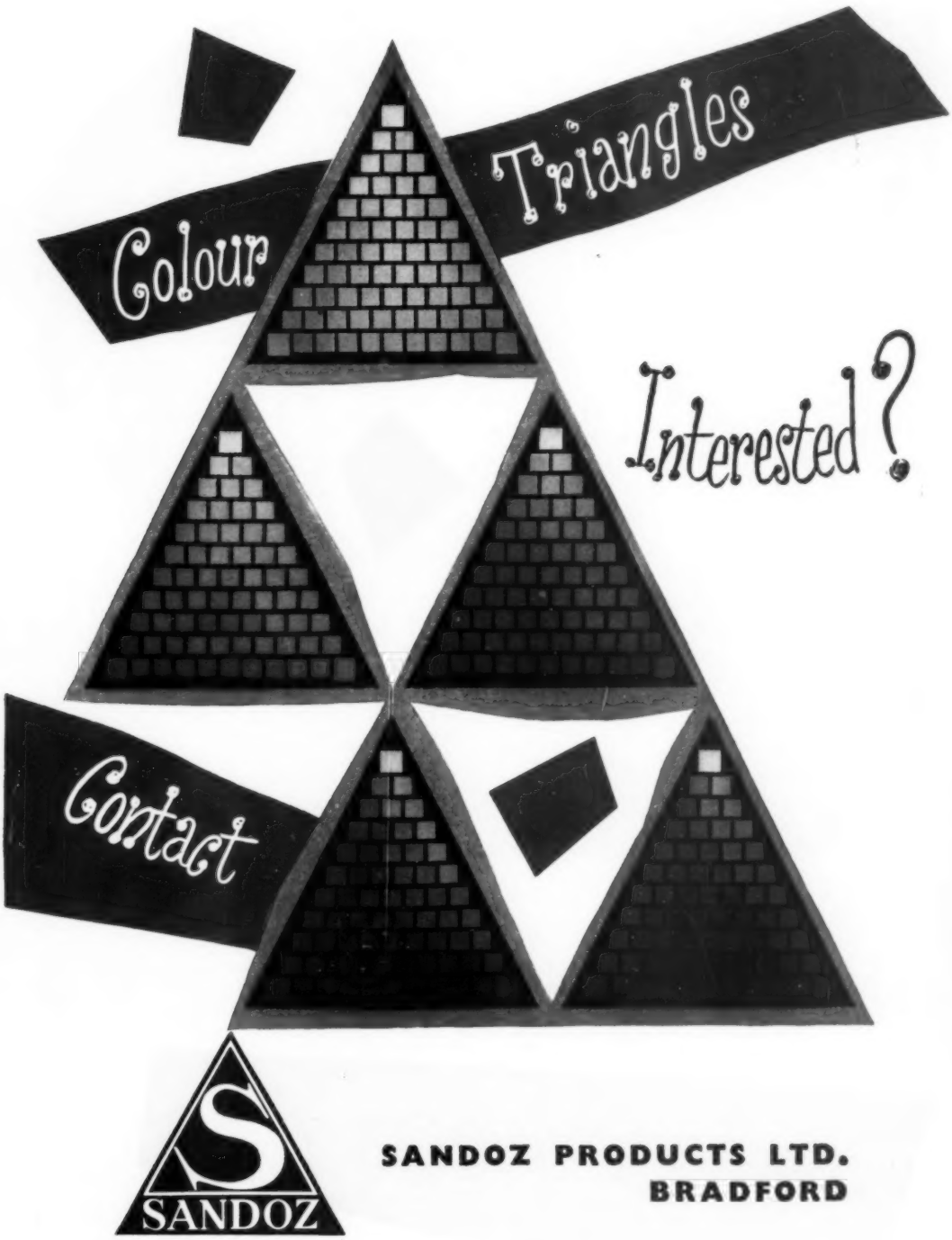
MANCHESTER SECTION. Annual General Meeting. 7 p.m. To be followed by a paper entitled *Bleaching of Cotton and Cotton Blends with Hydrogen Peroxide.* G. C. Woodford, Esq. (Laporte Chemicals Ltd.). The Textile Institute, 10 Blackfriars Street, **Manchester 3.**

Friday, 25th April 1958

The Society's Annual General Meeting and Dinner. The Central Hotel, **Glasgow C.1.**

Friday, 2nd May 1958

LONDON SECTION. Annual General Meeting and Dinner. Aldwych Brasserie, Aldwych, **London, W.C.2.** 6 p.m. for 6.30 p.m. (Mr. C. O. Clark will speak on the standards required for the A.S.D.C. examination.)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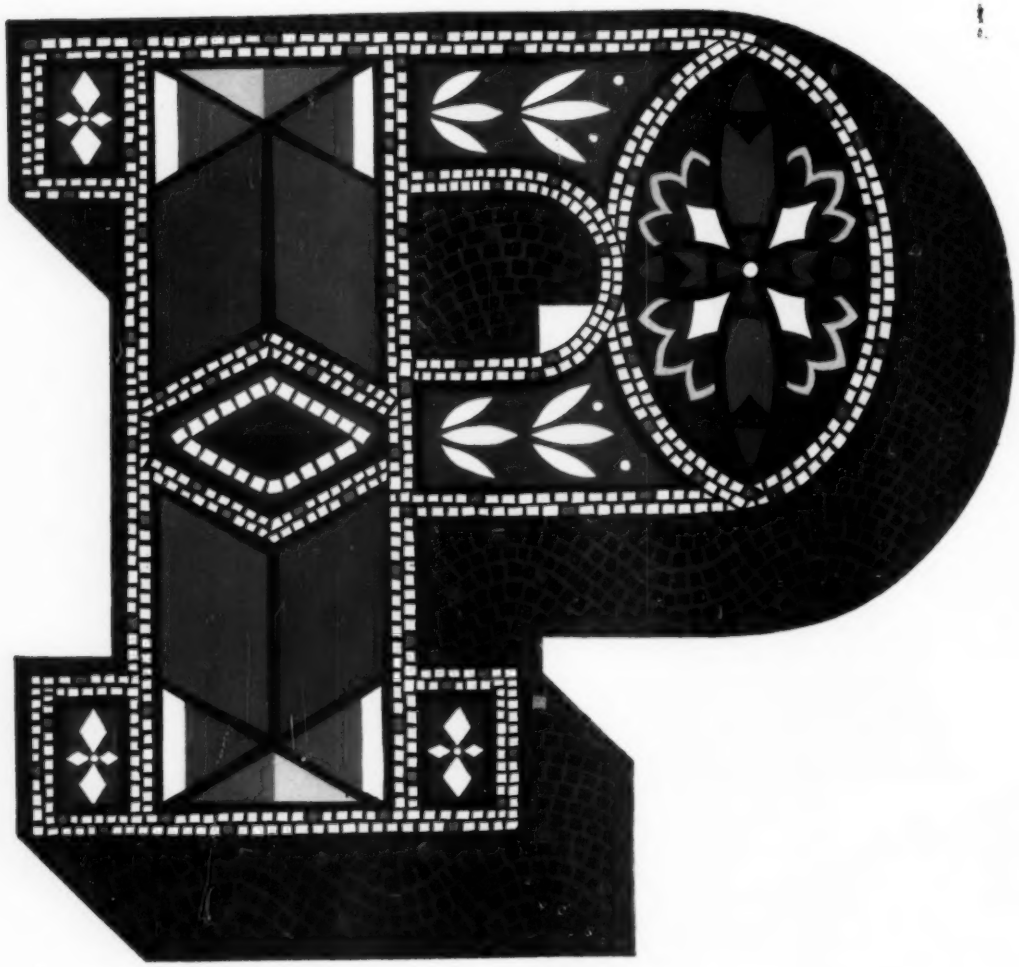

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THE JOURNAL

OF THE

Society of Dyers and Colourists

Volume 74 Number 2 FEBRUARY 1958 *Issued Monthly*

Proceedings of the Society

Determination of Performance of Fibre Blends in Wet Processing

R. C. CHEETHAM and H. D. EDWARDS

Meetings of the Midlands Section held at the Leicester College of Technology and Commerce on 23rd January 1957, Mr. C. A. Mills in the chair; of the Manchester Section held at the Textile Institute, Mr. J. Boulton in the chair; and of the Northern Ireland Section held at Thompson's Restaurant, Belfast, Mr. R. G. J. Reid in the chair

The behaviour of fibre blends during wet processing such as scouring, milling, and dyeing has been studied with the object of developing laboratory techniques to simulate works conditions. A method is described for determining the behaviour of fibres under pack-dyeing conditions. In the case of fabrics, particular attention has been given to the effect of processing strains on dimensional stability.

It has been demonstrated that the temperature–extension curves for fabrics from different acrylic fibres show characteristic points of inflexion. Apparatus for measuring fabric crease recovery in hot liquors is described, and the information obtained has been used to study rope marking in winch dyeing.

Milling has received attention with reference to feltmaking, and an experimental method of evaluation has made use of small-scale machines for carding and milling. Using this technique, the effect of acid liquors of various kinds in felting has been measured. Fabric has also been used in the small-scale milling stocks to obtain the optimum conditions for milling a Fibrolane–wool velour cloth.

INTRODUCTION

As modern textiles continue to make use of mixed fibres and blends, it becomes increasingly necessary to consider means of assessing the behaviour of yarns and fabrics during dyeing and finishing. Whilst a considerable amount of data is being collected by dyers and finishers, often working in collaboration with dyemakers and fibre producers, efforts are usually concerned with the treatment of specific examples of blended fibres rather than with a comprehensive attack on the general behaviour of fibres in wet processing. If the large number of new fibres being developed, and hence the almost unlimited permutations possible, are borne in mind, it will be appreciated that, without a systematic approach to the behaviour of fibres during wet processing, blend dyeing could conceivably be difficult and costly, since often the problems involved would have to be dealt with on a commercial scale. In practice the dyer and finisher now has to solve most of his problems by trial and error, and then eventually reach a compromise between conditions to suit the various types of fibre in the blend being processed.

In our approach to these problems we have worked with small-scale equipment and have attempted to correlate the effect of wet processing on fibre properties with the probable changes of fabric or yarn in bulk processing, particularly from the point of view of dimensional stability. It has been a basic principle to devise, as far as possible, methods and apparatus simulating the conditions expected in bulk working.

It is not proposed to concentrate on the details of the results obtained but to describe the techniques we have developed for correlating fabric performance with fibre properties.

"Wet processing" is the general term used to cover the operations involved in preparing for sale by removing spinning oils and foreign matter, by stabilising a fabric through releasing loom strains and consolidating the structure, by applying colour, and by preparing the surface.

We have therefore to consider not only the effect of the chemical composition of the various baths and the temperatures used, but also the effect of mechanical action under these conditions. While the behaviour of fabrics containing one component might be deduced if there were sufficient data of the right kind available, fabrics which contain more than one fibre must be considered empirically, since at the present time we have insufficient knowledge of the strains carried by the individual components in the engineering of blend fabric structures, nor have we knowledge of the way in which these stresses are affected by alterations in

processing conditions. Among our objects have been to find practical ways of simulating the stresses imposed by some of the treatments and to examine the reaction of various types of blend to processing liquors. The processes so far considered have mainly been concerned with winch dyeing and milling, which are both of fairly general application in textile finishing. In our approach we have had to consider that, in general, blends will have to be processed on conventional machinery, although one of our objects has been to consider possible methods of modifying this machinery.

This study is intended to simplify the picture, so that we may have a basis for designing the procedures for bulk work.

CONSIDERATION OF STAPLE PROCESSING

Let us consider the very simple case of dyeing a fibre in a cage machine of the Obermaier type. We obviously have to choose dyebath conditions which will give economic exhaustion, an effective dyeing rate, and no degradation of the fibre. We must also achieve an economic machine loading and ensure that the load in the machine will be so arranged as to give a sufficient liquor flow without channelling and thereby achieve even penetration and level dyeing.

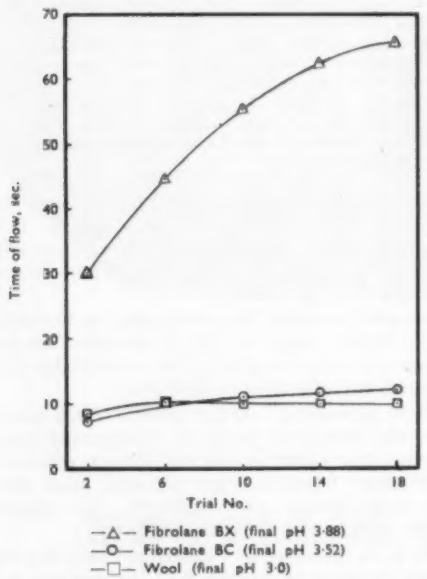


FIG. 1— Formic Acid Solution (4% on weight of material at liquor ratio 10 : 1) at 100°C. for 1 hr.

The reaction of the fibre will be determined by its staple length and denier, by its crimp and resilience, and by the way in which these latter properties alter on wetting out and during the dyeing process. It is well known that loose wool presents few dyeing difficulties because of its ability to maintain resilience when wet and therefore to keep an open character during dyeing.

The resilience of a fibre during pack dyeing has been examined in a simple way by measuring the rate at which a known volume of hot liquor flows through a weighed quantity of fibre formed into a pad of diameter approx. 2½ in. and thickness ½ in. The experiment is repeated several times, and the time of flow plotted against the cycle number. A fibre of good characteristics will maintain a constant rate of passage of liquor as the cycles are repeated, and some typical results are shown in Fig. 1. Wool shows, under this test, its typical quality of maintaining its resilience when hot and wet, and consequently we would expect wool to have a beneficial effect in most forms of package; we have this in practice, as is shown by the packages of blend yarns containing wool. Hence we might expect the inclusion of wool in a blend in the unusual rôle of a "diluent" on account of its contribution to ease of wet processing.

This property of wool, viz. its ability to withstand compression forces when wet, is of very great importance in cloth finishing, and its property of recovery from stretching forces has similar significance.

The case of package dyeing is used as an introduction because it does show that the fibre behaviour under certain practical conditions is of paramount importance. Our experiments also showed closer correlation with practical performance than would have been expected from single-fibre properties. Equally, we found that incorrect dyebath conditions were shown by a loss in fibre properties, the filtration rate being a very sensitive test. In a test of this type, which can be extended also to blends of staples, the fibre arrangement is random, and no structural features have to be considered.

BEHAVIOUR OF FIBRE BLENDS IN FABRICS

In the past, much work has been done on the types of dyebath or other processing solutions which should be used for different fibre blends. As with package dyeing, this work has had as its object the provision of suitable conditions for economic dyeing. However, the presence of more than one fibre has usually meant the adoption of compromise conditions, not only because of the different classes of dye used but also because of the necessity for avoiding chemical degradation of the different fibres in the blend.

It is axiomatic that the performance of a finished fabric depends on its processing history, and if chemical degradation of the fibres has occurred, this fault will be reflected in the finished state and possibly during wear. However, during its processing history the fabric will have been subject to various types of mechanical action, and all these influences will have contributed to the final fabric and its performance. In our work with blends we are finding to an increasing extent that mechanical movement is the aspect which demands most attention if we are to solve the problems of making stable fabrics of desirable handle and appearance.

If we look back to see how conventional machinery has been modified to cope with blends, we find that the wool–cellulosic fibre blends have usually been processed on conventional machinery with minimum alteration. There are two factors which have operated, the first being that, since wool is the "premium" fibre, the cloths have often been designed to have more of the wool fibre at the surface, and the second that during wet processing there is a tendency for the wool fibre to migrate to the surface. Consequently, it has often been possible to use conventional wool machinery to produce the required finish without having to consider the special requirements of the other fibre. However, with some blends of wool and cellulosic fibres difficulties have arisen owing to the severe mechanical action of heavy wool machinery on the cellulosic fibres in the swollen condition, and also certain structures have lacked dimensional stability after wet processing.

With such blends there is therefore an obvious need for more precise information, and it is worth examining the effect of processing strains on hot wet materials. This, too, has been studied in a simple manner.

FABRIC EXTENSION AND RECOVERY— EXPERIMENTAL

Strips of fabric are prepared, and two reference marks made a fixed distance apart. Enough strips are taken to cover a required range, and each is loaded with a known weight and then suspended under a processing solution at the boil for 1 hr. The strips are then measured again, and the extensions noted and plotted against load/cm. The strips are allowed to dry without tension, remeasured, and then relaxed in soap solution at 60°c. and measured again. After drying again, the relaxation procedure is repeated until no further change takes place. The results are then expressed on a graph.

The acrylic fibres used in our experiments were various commercial products which were available at the time.

This type of experiment shows how final fabric dimensional stability is related to the tension imposed during processing, and Fig. 2 illustrates how sensitive to extension is a fibre of low wet tensile modulus and how difficult it is to achieve fully relaxed stability of a blend containing such a fibre after excessive handling. The successful processing of such fabrics depends on applying only minimum tensions throughout, so that the stresses do not give extensions beyond the limits of recovery in a single drying cycle.

With thermoplastic fibres, such as the acrylics, similar extensions occur, but the fabrics do not relax in the same way as those containing swellable fibres (see Fig. 3). We would expect this type of extension from a study of the variation of initial tensile modulus of the single fibre, which shows how this falls with increasing temperature. Single-fibre measurement will not, however, give information on the performance of a blend. Fig. 4 shows how the presence of a blend fibre (wool) limits extension. This type of experiment can be used to determine the proportion of a blend fibre to be added to an acrylic fibre to assist in carrying the fabric through wet processing.

A further important property which must be studied with those fibres which are heat-sensitive

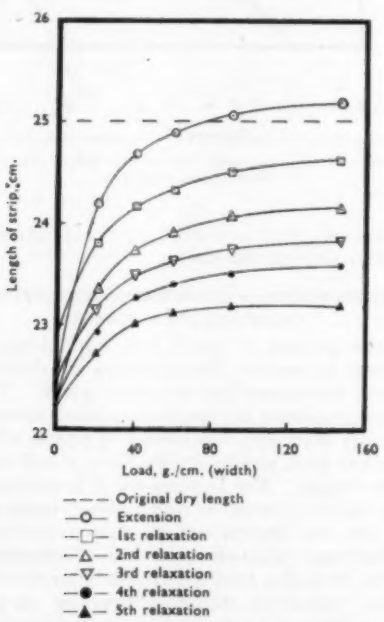


FIG. 2— Fabrics from Blends of Fibres having Low Initial Tensile Modulus

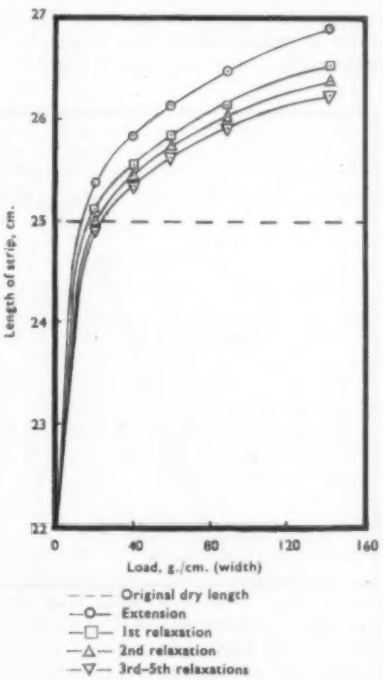


FIG. 3— 100%-Acrylic Fabric

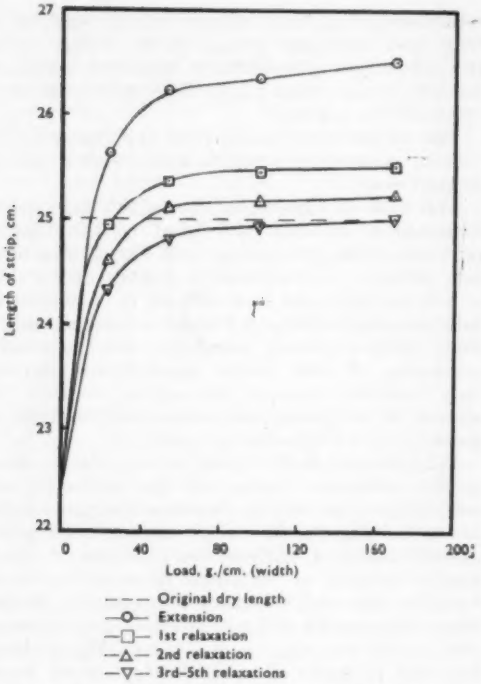


FIG. 4— Fabric from 50% Acrylic–50% Wool Blend

is the variation of extensibility with temperature. This is obtained by suspending in a processing bath a strip of fabric with a known load attached and following by means of a cathetometer the extension as the temperature is raised to the boil. Some typical results are shown in Fig. 5–7. This type of experiment is important in that it shows various transitions which occur and which must be taken

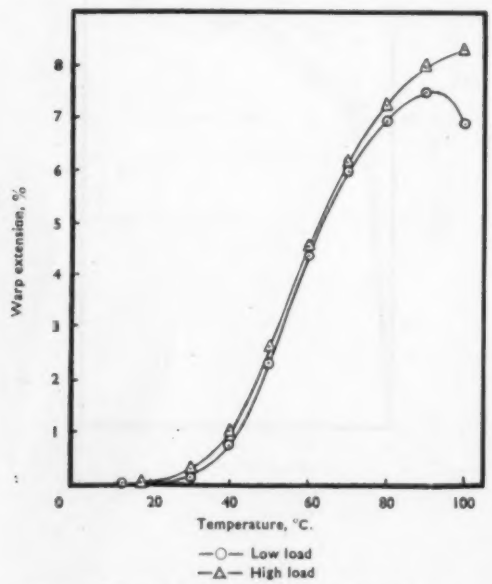


FIG. 5— 100%-Acrylic Fabric having Basic Dyeing Properties

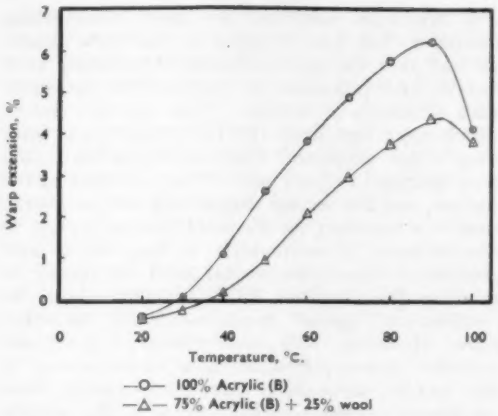


FIG. 6— Fabric from Acrylic (B) with Wool-dyeing Properties and from Blend with Wool

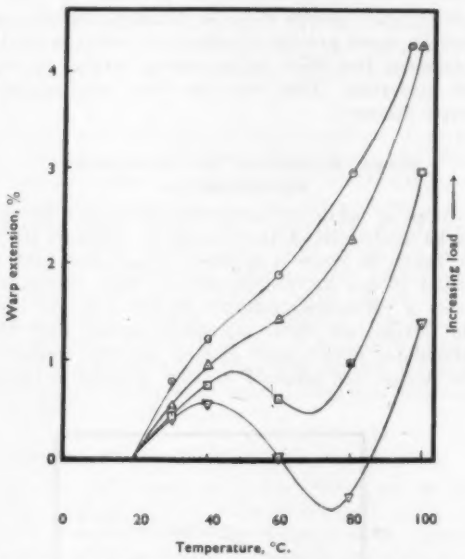


FIG. 7— Fabric from 60% Wool + 40% Acrylic (A) with Wool-dyeing Properties

into account if it is desired to avoid introducing irregularities into the cloth.

ROPE MARKING OF FABRICS CONTAINING THERMOPLASTIC FIBRES

Before putting a fabric into rope form, it is necessary to remove latent strains by relaxing in the hot wet condition in open width. This is because processing in rope form depends on random formation of creases, and release of strains after the fabric has been put into rope form would tend to set the creases. For this reason it is necessary to know whether there is any critical temperature (this can be determined as shown above) and whether any time factors are of significance. Further, it is also desirable to discover whether or not the relaxation shrinkage depends on pH, as there is little point in giving a relaxation at the boil at pH 7 if further shrinkage is going to occur

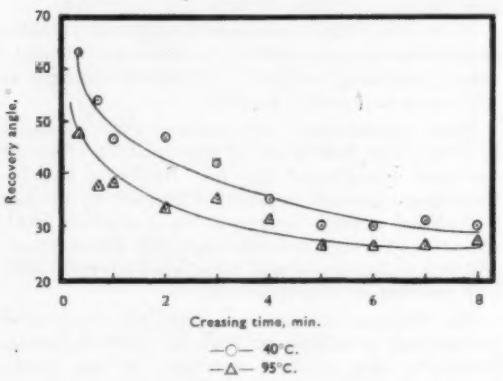
in the dye vessel at, say, pH 3. It is also desirable to know how long it will take to achieve complete relaxation: with swellable fibres the process is rapid, but with hydrophobic fibres an appreciable time is taken. The information required is obtained very simply by measuring warp and weft shrinkage at the boil and plotting against time over the pH range investigated.

Thermoplastic and thermosetting fibres, as we have seen, do not easily recover from deformation when hot and wet, and consequently even in blends they are particularly subject to faults such as rope marking during winch dyeing. This rope creasing may occur even though all loom strains have been released, and is particularly difficult to study on the laboratory scale and very costly to study in the works. Also, assessment of results is purely subjective.

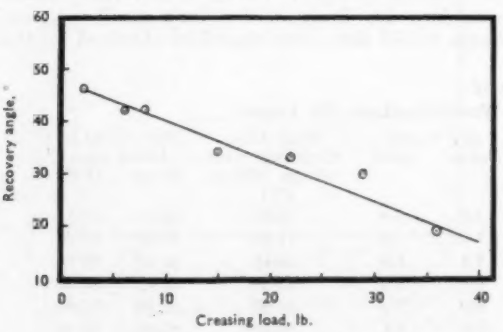
There is, however, a further type of measurement which can be made and used for the elimination of such faults. This has been applied to Tricel–wool worsted cloth.

During winch dyeing, the fabric is creased on the reel and then moved through and out of the liquor, during which time the creases have the opportunity of opening out.

It seemed likely that the measurement of crease recovery in hot liquors might be of interest in the study of these rope-marking problems. Accordingly, a method was devised by the Droylsden Physics Laboratory for providing these measurements (see p. 84), and Fig. 8 shows some of the results obtained. It will be seen that the crease recovery in hot liquors is very sensitive to creasing time and is also related to creasing load. It is easily understandable, therefore, that the technological solution to rope creasing in this particular cloth was largely achieved by speeding up the winch and so reducing the creasing time, and also by lowering the winch reel and jockey roller, thus reducing the creasing load. It seems that improvements in winch design will follow as information is collected on the strains imposed by the machinery on fabrics of different construction and is correlated with the laboratory study of the effects of such loads on final fabric performance. However, it should be emphasised that the static loads used in our experiments can give only a general picture, and it is also necessary to devise some experimental method which will enable measurements to be made of repeated stresses acting for a very short time.



A— Effect of Creasing Time (creasing load 10 lb.)



B— Effect of Creasing Load (creasing time 5 min. at 95°C.)

FIG. 8— 60% Tricel–40% Wool

EFFECT OF MILLING ON FIBRES

While in dyeing the effect of mechanical action on the fibre is incidental but unavoidable, in the case of milling mechanical action is deliberately applied in order to achieve the consolidation of cloth structure. Milling merits particular attention owing to its widespread application to the production of felt and fabric structures, and a suitable laboratory technique has had to be devised.

One approach in this investigation was to use as experimental material some flat felt produced by carding small weights of fibres in a hand card and then consolidating the web so formed on a plate hardening machine as used in the felting industry. As little as 4 oz. can be fed into the hand card, and this quantity would produce flat felts measuring approx. 20 in. × 16 in. The pieces of felt can then be cut into convenient squares for a series of experiments in the Furness milling stocks, which, again, are capable of handling small weights of material. With this technique, a useful indication was obtained of the effect of various acid milling liquors on felt strength.

Experimental

In one series of experiments designed to measure the effect of various acid liquors on a blend of Fibrolane BX and wool compared with all-wool material, eight flat pieces of felt were made from the blend and from wool, as described, and each piece was cut into sixteen pieces measuring 6 in. × 6 in. Two pieces from each flat were then combined for one experiment, thus ensuring that possible differences between various flats were eliminated.

During the actual felting operation in the milling machine, a technique was adopted to produce and maintain regularity of shape so that area shrinkage could be recorded at intervals by measuring the length and the width of each square. The felting was continued until the size of the squares was reduced to 4 in. × 4 in., at which stage the time of milling was noted. Patterns were then

cut from each milled square and tested for thickness on a Mercer thickness gauge, and tensile strength on the Denison testing machine. The data so obtained were used to give some indication of felt quality.

As Table I shows, a blend of wool and Fibrolane mills at a quicker rate than wool, but the time required to mill to final dimensions decreases with fall in pH in both cases. Moreover, it was observed that physical properties such as density and tensile strength were a maximum when milling was conducted at a lower pH value, more particularly in the case of the blend.

TABLE I

Milling in Acid Liquors

Acid Solution	pH Value (start)	Time of Felting (min.)	Tensile Strength (kg./sq.cm.)	Density (g./c.c.)
80% WOOL–20% FIBROLANE BX* FELT PATTERNS				
Sulphuric ...	1·45	53	30·09	0·1929
Phosphoric ...	1·78	58	28·90	0·1834
Acetic ...	3·90	77	26·79	0·1721
Lactic ...	3·85	78	26·39	0·1704
100%-WOOL FELT PATTERNS				
Sulphuric ...	1·62	67	27·63	0·1618
Phosphoric ...	2·00	75	27·03	0·1608
Acetic ...	3·90	87	26·21	0·1609
Lactic ...	3·94	86	26·51	0·1548

* *Fibrolane* is a registered trademark of Courtaulds Ltd., used to denote its regenerated protein fibres.

This method uses the same approach as that used to study the behaviour of fibres during package dyeing; i.e. the results are obtained on a random arrangement of fibres. Thus milling can be studied from the point of view of its effect on fibres as distinct from the effect on a fabric, where often the course of milling shrinkage is profoundly affected by several factors, such as number of warps and weft threads, twist and counts of yarn, and also by the weave. Fig. 9 shows how milling rate is affected by difference between plain and twill, and the angle of twill.

Fabric was, however, used, bearing these qualifications in mind, in a series of experiments with the object of discovering a modified milling liquor for a cloth made from yarns of relatively low tensile strength or from yarns having insufficient resistance to milling liquors containing strong alkalis such as soda ash.

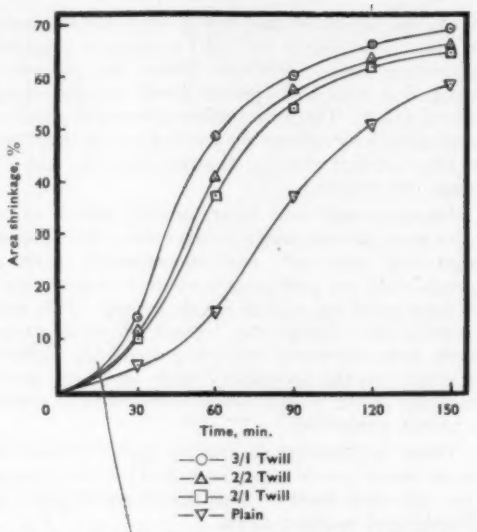


FIG. 9— Area Milling Shrinkage of Cloths of Different Constructions

For example, a velour cloth made from warp and weft blend yarns composed of 70% wool and 30% Fibrolane BX was given a variety of milling treatments as outlined in Table II, and periodically the cloth was measured for shrinkage and finally for tensile strength. In this experiment the area shrinkage was measured after a fixed time corresponding to a normal cloth-milling treatment.

It was found that a mixture of soap and synthetic detergent was most effective in producing adequate rate of shrinkage coupled with highest strength as well as an acceptable handle.

These preliminary experiments were intended to give some indication of the relative effect of variously constituted liquors. Applying the information obtained, we found it possible to develop a modified milling technique for a woollen cloth constructed from wool–Fibrolane BX blend yarns, and this technique is now being used commercially in a number of constructions.

The Furness milling stocks have also been used to evaluate a milling method for knitted berets. Normally, the smallest machine of the push-bumper type, used for milling berets, would accommodate approx. 100 lb. of material, and no machines are designed for very small weights. Some useful data were therefore obtained on the

TABLE II

Effect of Milling Liquors on Wool–Fibrolane BX Fabric

Milling Solution	pH Values Before	pH Values After	Mean Area Shrinkage after 60 min. Milling (%)	Mean Load to Break (kg.) Warp	Mean Load to Break (kg.) Weft
Acid milling (3% sulphuric acid)	1·3	5·0	23·50	26·21	31·12
0·2% Lissapol N (ICI)	7·0	6·6	21·90	29·39	33·29
0·2% Lissapol N + 3% sodium chloride	7·1	5·9	24·10	31·43	36·65
0·2% Lissapol N + 3% sodium bicarbonate ...	8·4	8·9	25·60	27·62	30·39
0·1% Lissapol N + 0·1% soap	8·9	7·0	28·36	32·66	35·65
0·5% Soap	10·0	7·1	26·80	29·76	35·30
0·5% Soap + 0·2% sodium carbonate	10·7	8·4	26·25	27·26	28·71
0·5% Soap + 0·5% sodium carbonate	11·1	8·8	22·50	23·67	27·12

composition of milling liquors by treating two berets at a time in the Furness milling stocks. The usefulness of this machine could no doubt be extended still further by attaching a steam spray and by totally enclosing the machine so as to operate at higher temperatures and to prevent heat losses during experiments.

The type of experiments described to trace the course of felting can, of course, be used to test the efficiency of non-felting treatments and of blends of fibres to give non-felting characteristics.

Relative Effect of Milling on Cloths composed of Various Fibres

In milling, mechanical action is required in order to achieve the consolidation of cloth structure, and it may be of interest to show the behaviour of three selected fabrics of the same construction but containing different fibres. Fig. 10 shows the effect of Fibrolane BX in increasing the milling rate of wool and the effect of an acrylic fibre, which almost completely inhibits milling shrinkage. If milling is continued in this case, the wool works to the surface and is shed as flock.

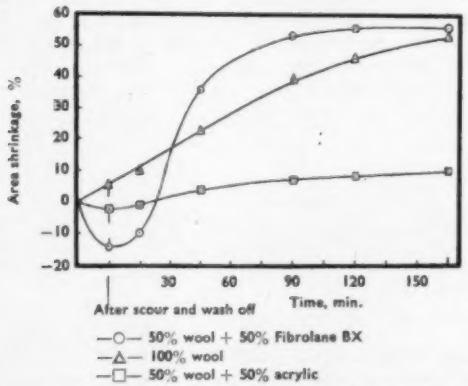


FIG. 10— Rate-of-milling Curves for Velour Cloths— Soap Milling

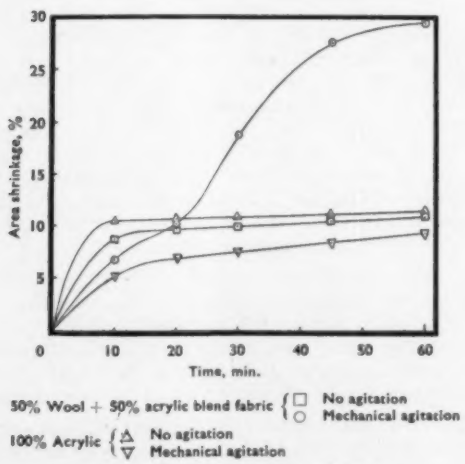


FIG. 11— Area Shrinkage in Boiling Water

Although acrylic fibres prevent milling shrinkage under normal processing conditions, this is not the case if the acrylic fibre is brought into a plastic condition. Fig. 11 shows the behaviour of an acrylic fibre as 100% fabric and in blend with wool on shrinkage in boiling water, when we see that in the presence of wool, milling does take place if mechanical action can be applied while the acrylic fibre is in a plastic state.

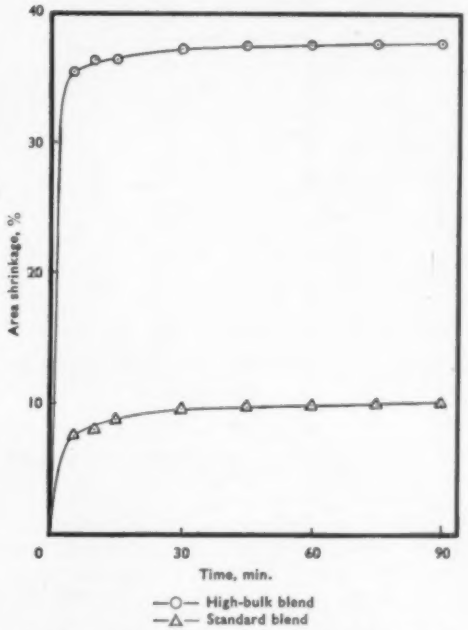


FIG. 12— Area Shrinkage of Acrylic–Wool Blend Fabrics

Another approach to the construction of dense fabrics is the use of high-shrinkage acrylic fibres in blends. Fig. 12 shows the large amount of shrinkage which can be obtained. By such means it may be possible to depart from traditional heavily milled constructions by using blends of normal with high-shrinkage acrylics, of high-shrinkage acrylics with, say, wool, and by using different bulking yarns in warp and weft. In this case, for blends with wool it might be desirable to give a slight milling to produce cover and to lock the fibres.

CONCLUSION

From the simple experiments described, it is possible to foresee the types of problem that are likely to occur in the wet processing of blend fabrics and therefore to select and to give attention to those topics requiring detailed treatment.

* * *

In this type of work, we are concerned with the way fundamental fibre properties affect the technology of fabric dyeing and finishing, and we are very glad of the opportunity to thank our colleagues of the Droylsden Physics Laboratory for their interest.

APPENDIX

Apparatus for the Measurement of Crease Recovery in Hot Liquors

J. C. GUTHRIE

Creasing is carried out with the fabric sample submerged in a liquid, and the recovery angle is measured with the sample still under the liquid surface.

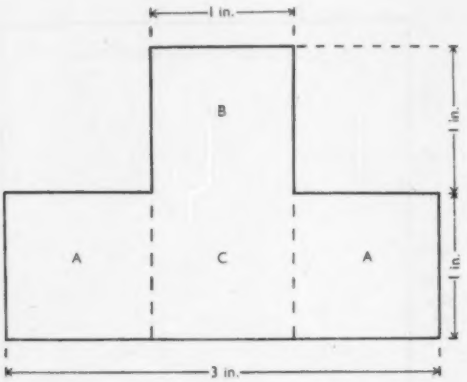


FIG. 13

Test specimens are cut as shown in Fig. 13. The tabs *A* are folded back and clamped in the holder, leaving tab *B* free. The holder with the specimen is then immersed in the thermostatically controlled liquid bath. One minute is allowed to elapse so that the sample can wet out, and tab *B* is then folded back over *C* to form the crease. A glass plate is placed over *B*, and a weight added for a given time. The weight is then removed, and the holder lifted into an upright position, so that the crease just formed is vertical. The specimen holder is beneath the liquid surface throughout, and is

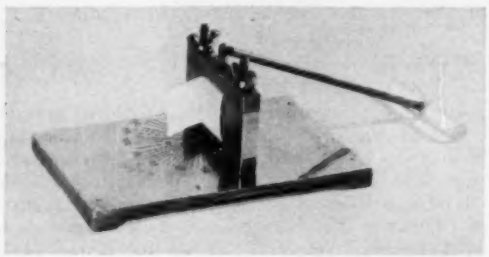


FIG. 14

easily manipulated from above the surface by means of its handle (Fig. 14). When the sample and the holder have been placed in a vertical position, the holder slips into slots in the base plate. The base plate carries a scale of degrees, and the recovery angle is read off (viewing through the liquid) from directly above.

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(*MS. received 20th June 1957*)

Discussion

MIDLANDS SECTION

Mr. C. A. MILLS: Is the lecturer prepared to enlarge on his statement that crease marks can be obviated by increasing winch speed? It has long been accepted by dyers that decreasing the speed is the effective way of dealing with the problem.

Dr. EDWARDS: The work that we did on the Tricel–wool blend showed quite conclusively that the crease recovery in hot liquors was very much better for short creasing times, and therefore the technological solution for the rope marking problem was found by increasing winch speed. The example of two Tricel–wool fabrics dyed in one case at a linear cloth speed of 30 and in the other at 90 yd./min. shows conclusively that the higher winch speed is to be preferred for this cloth. It is quite possible that for other types of blend fabric there would not be a corresponding increase in crease recovery at very short creasing times, and the improvement might be offset by other factors such as viscous drag from the liquid.

Mr. A. W. CARPENTER: It is well known that, in dyeing fabrics made from acrylic fibres, it is essential to cool slowly to avoid rope marks. The lecturer has suggested that it is also important to commence dyeing below the critical plastic temperature and to raise slowly to the boil. As it has been accepted in the past that creases are removed by boiling, why is this precaution necessary?

Dr. EDWARDS: Winch dyeing depends for success essentially on the random formation of creases during dyeing. If there is any non-uniformity in the fabric, then creases will be predisposed to form at certain points, and marks will therefore be set into the cloth. In the same way, if once the cloth is in rope form and it is subsequently deformed, there will be a tendency for the creases to reform in the same places. For this reason it is advisable to take great care in passing a fabric through transition points.

In reply to the second part of the question, while it is true that in some circumstances creases can be removed by further boiling, unlevel dyeing caused by static liquor held inside the creases will not be so easily removed, and therefore it is advisable to take care both in heating up and in cooling down.

Dr. A. DATYNER: The lecturer has suggested that new blends or fabric constructions be tested by the dyer according to the methods he has

outlined. It would appear that these tests are rather time-consuming. Would such routine be practicable in a commission dyehouse handling a wide variety of fabrics?

Dr. EDWARDS: The tests we have outlined were developed as a research tool to enable us to devise methods for the handling of different fibre blends. The apparatus is extremely simple and it is quite likely that a simplified system of testing would give sufficient information for a dyer to be able to anticipate the types of difficulty likely to be encountered.

Mr. MILLS: It is unfortunate that manufacturers often make fabrics without first ascertaining by such tests that they can indeed be processed to requirements. The dyer is simply informed that the cloth is already made, and is left to fight a losing battle, which brings both the maker and himself into disrepute.

Dr. EDWARDS: We have constantly been stressing in our lectures that it is in the interests of all parties that the earliest possible consultation be held between the manufacturer and the dyer, when considering the promotion of new blend fabrics, and the aim of the techniques we have described is to enable the dyer to make some forecast from a very small sample of the probable wet-processing limitations of a particular fabric blend or construction.

Dr. DATYNER: Does not the creeping of the wool, when blends of that fibre with acrylics are milled at low temperatures, seriously detract from the value of these mixtures for cloths subject to friction in wear, e.g. suitings?

Dr. EDWARDS: We illustrated in the lecture the movement of wool in blend fabrics under milling conditions, when fibre movement most definitely takes place. In normal types of cloth construction during dry wear it is unlikely that this migration would occur.

Mr. C. BRYAN: Blends of acrylics with wool are being prepared with varying proportions of the component fibres, and some examples I have seen have been very poor in handle. Is any information available on the optimum proportion of acrylic in these blends?

Dr. EDWARDS: The handle of a fabric depends not only upon the proportion of component fibres in the blend but also upon the techniques used in finishing. Furthermore, the effect of denier is very marked in producing changes such as crispness and drape. Acrylic blends can be used over quite a large range of proportions, depending upon which particular property of the fabric it is desired to stress.

COMMUNICATION

Measurement of Damage in Wool Materials

IV—A Modification of the Krais-Markert-Viertel (K.M.V.) Test—The Supercontraction of Keratin Fibres in Solutions of Caustic Potash

J. W. BELL, D. P. VELDSMAN, and C. S. WHEWELL

Dimensional changes of wool fibres when immersed in 20% caustic potash solution are studied and data are given showing the effects of various chemical pretreatments on the supercontraction and the time of recovery from supercontraction.

During the last few years increasing attention has been paid to the use of the K.M.V. reagent [1] (a concentrated solution of caustic potash saturated with ammonia) for assessing damage in wool materials. Modifications of the original test include those by Schirlé, Meyer, and Gianola [2] and by Rzegocinski and Lund [3]. These latter authors measure changes in fibre length rather than the time for bubbles to appear on the surface of fibres. Quantitative observations of changes in fibre length in the K.M.V. reagent are, however, somewhat inconvenient because of the escape of ammonia, and consequently the present investigation was carried out to examine the changes taking place when fibres are immersed simply in concentrated solutions of caustic potash.

EXPERIMENTAL

(1) *Purification of Wool and Related Materials*

All the fibres used in this investigation (Lincoln wool of diameter 45–50 μ. and mohair of 40 μ.) were purified by consecutive extraction with ethyl ether and alcohol followed by thorough rinsing in distilled water.

(2) *Measurement of Dimensional Changes in Fibres immersed in Caustic Potash Solutions*

A microscope was fixed so that its stage was vertical, and a glass tank having double walls on three sides was fixed to the stage. Water (usually at 28·5 $\pm$ 0·2°C.) from a thermostat was circulated through the space between the walls. Vertically above the tank, on the edge of the stage, a piece of flat cork with a small hole in the centre was fixed in a horizontal position with plasticine. Around the hole, on top and underneath, were placed two blobs of Plasticine to fix in position a thin glass rod, on the lower end of which the fibre to be examined was mounted. Caustic potash solution (5 ml.) was pipetted into the tank.

To mount the fibres, the following technique was employed. To one end of a rod (8 cm. long, 2 mm. in diameter) a small amount of sealing wax was fixed and drawn out to a point. The point was then melted in a microflame to form a bead, and the end of the fibre was quickly attached to the softened wax. The fibre was then cut to a length of 0·7–0·8 mm. The other end of the rod was pushed upwards through the hole in the cork, the

tank was moved downwards, and the rod was positioned and finally fixed with plasticine so that the fibre was just above the level of the alkali solution and in front of the condenser of the microscope. The tank was now moved upwards so that the fibre was completely immersed. A stop clock was then started and the fibre focussed.

TABLE I

Experiments on Lincoln Wool and Mohair*

Concn. of KOH (%)	"Strike" (min. sec.)	Super-contraction (%)	Time to Max. (min. sec.)	Recovery Time (min. sec.)
5	2 20	14	6	14–15
10	1 30	17 (20)*	3	4 2 (3 5)
15	1 20 (55)*	19 (20)	2 20 (1 55)*	3 12 (2 22)
20	1 0 (40)	35 (35)	2 50 (1 30)	2 0 (2 5)
25	1 20 (30)	35 (34)	—	3 10 (2 20)
30	1 20 (30)	35 (33)	—	3 42 (3 10)
31	1 20	35	—	4 0
32	1 15	38	—	5 0
33	1 20	36	—	6 0
35	1 40	37	—	7 0

* Figures in brackets refer to mohair.

Preliminary observations had shown that the supercontraction of the fibre might not be the most suitable parameter to measure. Consequently, measurements were also made of the time at which the fibre started to contract, and finally the time at which it recovered its original length (owing to swelling). The sequence of making the determinations was as follows: The length of the fibre was measured on a graticule scale in the eyepiece of the microscope. Then the time at which the fibre started to contract was noted (this is referred to subsequently as the "strike"); thereafter the maximum supercontraction, and finally the time taken for the fibre to return to its original length, were recorded. If the fibre did not recover completely from supercontraction after 6 min., the percentage recovery was noted. The recovery time refers to the interval between the attaining of maximum contraction and recovering the original length.

RESULTS AND DISCUSSION

(1) *Dimensional Changes in Caustic Potash Solutions of Various Concentrations*

Both Lincoln wool and mohair were used in these experiments, and the values of the various characteristics mentioned previously were determined for fibres placed in caustic potash solutions of various concentrations. The results obtained are summarised in Table I. Each value represents the mean of at least twenty determinations.

From the results in Table I it can be seen that the supercontraction attains a limiting value in solutions containing about 20% of caustic potash. In addition, the recovery time approaches a minimum value in solutions of this concentration. It would appear, therefore, that the 20% solution was the most suitable for use in the present work. It was, however, decided to use 30% solutions and not 20%, because the slightly increased time available for making the measurements facilitated obtaining accurate results.

(2) *Dimensional Changes of Modified Wool when immersed in 30% Caustic Potash Solution*

(i) ALKALI-MODIFIED WOOL— Data obtained on wool treated with 0·1 N. caustic soda solution at 30°C. for various times are summarised in Table II. In all cases the treated fibres were allowed to stand overnight in distilled water before being examined.

TABLE II

Time of Treatment (hr. min.)	Strike (min. sec.)	Super-contraction (%)		Recovery Time (min. sec.)
0	1 20	35		3 42
10	1 15	34		4 40
1 30	1 25	33		>6
2 0	1 20	34		>6
3 30	1 30	29	after	
16	1 30	21		>6
21	1 20	21	6 min.	
28	1 20	23		
48 (at 5°C.)	1 20	50		

It can be seen from Table II that the amount of supercontraction is not reduced unless the time of treatment exceeds 3·5 hr. This is presumably due to the comparatively slow formation of C–S–C linkings. Cross-link formation of this sort presumably takes place still more slowly at 5°C., and consequently treatment with 0·1 N. caustic soda is largely one of degradation, which accounts for the high supercontraction of 50% obtained on fibres treated for 48 hr. at 5°C. The recovery time is more rapidly affected by the treatment with caustic soda at 30°C., for even 10 min. treatment increases the recovery time from 3 min. 42 sec. to 4 min. 40 sec., while more prolonged treatment makes the fibres incapable of recovering within 6 min. This may be due to the presence of C–S–C or similar linkings. The strike, however, does not seem to be affected by the alkali treatment.

(ii) ACID-MODIFIED WOOL— Lincoln wool fibres were boiled in 0·1 N. hydrochloric acid for various times, then thoroughly washed in distilled water, and finally examined as described previously. The results are summarised in Table III.

TABLE III

Acid-modified Wool

Time of Boiling in Acid (hr.)	Strike (min. sec.)	Super-contraction (%)	Recovery Time (min. sec.)
Control	1 20	35	3 42
0·5	1 0	30	2 55
1·5	0 45	28	1 55
2·0	45	30	1 58
1·0 (0·5 N. acid)	25	28	1 10

It can be seen that, as the time and the severity of the treatment increase, the strike and the recovery time decrease. The supercontraction, however, remains substantially unaltered.

(iii) WOOL TREATED WITH DINITROFLUOROBENZENE (D.N.F.B.)— Lincoln wool (0·2 g.) which had been adjusted to pH 4·5 was wetted out in 5 ml. of a 4% aqueous solution of sodium bicarbonate. D.N.F.B. (0·2 ml. dissolved in 10 ml. of ethanol) was then added, and the mixture allowed to stand at 40°C. for 48 hr. The sample was subsequently washed thoroughly in several changes of distilled water and ethanol. Experiments were also carried out using different amounts of D.N.F.B. The results obtained on fibres treated in various ways are given in Table IV.

TABLE IV

Modification with D.N.F.B.

Concn. of D.N.F.B. (g./100 ml.)	Strike (min. sec.)	Super-contraction (%)	Recovery Time (min. sec.)
0	1 20	35	3 42
0·12	1 20	42	>6
0·46	1 20	36	>6
2·34	—	0	—

The striking features of these results are the inability of fibres which have been heavily treated with D.N.F.B. to contract and of any of the partially deaminated fibres to recover from contraction.

It was also found that fibres deaminated with the Van Slyke reagent* supercontract by 38% but do not recover their original dimensions. Blisters appear on the surface of these fibres almost as soon as they are immersed in the alkali.

(iv) CHLORINATED WOOL FIBRES— Fibres were chlorinated under both acid and alkaline conditions. For acid chlorination 0·1 g. of wool was wetted out in 3 ml. of water to which 0·02 ml. of concentrated sulphuric acid was added. Sodium hypochlorite solution (0·04 ml.) containing 10% by weight of active chlorine was added, and the fibres were allowed to stand in the mixture for 1 hr. The wool was then rinsed in water, immersed in 1·5% sodium bisulphite solution for 15 min., and finally rinsed overnight in running water. The procedure for alkaline chlorination was the same, except that 3 ml. of a 2% solution of borax was used instead of water, and no acid was added. The results obtained when the fibres treated in this way were examined are given below in Table V.

* Prepared by dissolving 8·62 g. of sodium nitrite and 7·1 ml. of glacial acetic acid in a few millilitres of water and making up to 20 ml. with water.

TABLE V

Chlorinated Wool

Conditions of Chlorination	Strike (min. sec.)	Super-contraction (%)	Recovery Time (min. sec.)
Control	1 20	35	3 42
Acid	40	40	4 52
Alkaline	58	38	6

The differences between the fibres are not marked, but the alkaline hypochlorite increases the recovery time, although both give similar values for supercontraction and strike.

(v) OXIDISED WOOL FIBRES— Lincoln wool fibres oxidised in several ways were examined, and the results obtained are shown in Table VI.

Although some marked differences are evident, it is clear that oxidation causes no consistent change, the variations depending on the type of oxidation.

(vi) REDUCED WOOL FIBRES— (*a*) *Reduction with Thioglycollic Acid.* 0·25 ml. of 90% thioglycollic acid was added to 4·5 ml. of water, and the pH was adjusted to 4·5 by adding a concentrated solution of potassium hydroxide. Lincoln wool and mohair fibres were treated in this solution (0·1 g. of fibre to 4·5 ml. of solution) at 30°C. for various times, and then rinsed overnight.

(*b*) *Reduction with Formosul.* Lincoln wool fibres were immersed for different times in a 1% aqueous solution of Formosul at 95°C., and then rinsed overnight. Results are shown in Table VII.

TABLE VI

Reagent	Conditions of Treatment	Strike (min. sec.)	Super-contraction (%)	Recovery Time (min. sec.)
Control	—	1 20	35	3 42
1% Peracetic acid	20 min. at 20°C.	12	38	1 5
	16 hr. at 20°C.	Immediate	19	22
1% Periodic acid	6·5 hr. at 20°C.	Immediate	30	50
0·5% Chromic acid	6·5 hr. at 20°C.	60	33	5 30
5-vol. Hydrogen peroxide at pH 10...	1·5 hr. at 50°C.	40	40	5 30

TABLE VII

Reduced Fibres

Time of Treatment (hr.)	Strike (min. sec.)	Super-contraction (%)	Recovery Time (min. sec.)
THIOGLYCOLLIC ACID			
Control	1 20	35	3 42
0·5	32	37	1 55
1	25	38	1 35
5	8	28	55
20	Immediate	29	1 5
FORMOSUL			
Control	1 20	35	3 42
10 min.	45	33	4 10
30 min.	35	30	Incomplete recovery

The data given in Tables II–VII indicate that the changes in dimensions and time of recovery of fibres in caustic potash are affected by the previous history of the fibre. For example, alkali-damaged fibres and untreated fibres supercontract by about 35%, but the recovery times of the alkali-damaged fibres are greater than those of untreated wool. Pretreatment with acid, however, reduces the supercontraction slightly, and the recovery times are markedly lower than those for untreated wool. Other reagents also affect both supercontraction and recovery time, although the changes in recovery time are much more marked: e.g. treatment with dinitrofluorobenzene, acid and alkaline chlorination, chromic acid, alkaline peroxide, and Formosul increase recovery time. Acid pretreatment, reduction with thioglycollic acid, and oxidation with peracetic and periodic acids reduce recovery time. Changes in the supercontraction in caustic potash caused by these treatments are comparatively small. It is, therefore, evident that if caustic potash is used in the K.M.V. test as modified by Rzegocinski and Lund, measurements of recovery time are to be preferred to those of supercontraction. Under the conditions described in this paper, it is possible to distinguish between acid and alkaline damage by determining the recovery time in caustic potash.

The data show, however, that many reagents other than acids and alkalis affect the changes which take place. The results of a test based on dimensional changes in caustic potash must be interpreted with great reserve, as must those obtained by the conventional K.M.V. test.

* * *

One of the authors (D.P.V.) wishes to acknowledge the financial assistance given by the South African Wool Textile Research Association.

Department of Textile Industries
The University
Leeds 2

(*Received 16th May 1957*)

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[1] Krais, P., Markert, H., and Viertel, O., *Forsch. Inst. Text. Ind. Dresden*, Forschg. H.14 and 15 (1933).
[2] Schirlé, C. H., Meyer, O., and Gianola, G., *J. Textile Inst.*, **47**, P 486 (1956).
[3] Rzegocinski, M. Z., and Lund, R., *ibid.*, **43**, T 91 (1952).

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Errors in the Assessment of Colour Fastness

Probably the majority of the members of our Society are interested in some way in the fastness properties of dyeings and therefore in the assessment of them. Thus papers such as the one in the *Journal* for October by Bülow and Horrdin[1] could be of considerable general interest, as it is well to know exactly what the assessments mean.

I feel, however, that the paper is perhaps not as helpful as it might be and, in fact, could give a misleading impression.

I must confess that I have little knowledge of statistical analysis, in which I expect I am in the good company of many other members, and therefore I should have welcomed a brief account of the method of calculating the components of variance, if that were possible, rather than to be referred to two publications. Also some details of Swedish Standard SIS 650013 could well have been included.

It appears that the method of making the light-fastness assessments is based on the American method, which is not favoured in this country. As a commentary on this method the paper may serve some purpose, but the statement is made that "the conclusions drawn should be valid also for other methods" and that is where, in my opinion, it may be misleading.

It is stated that blacks and blues are more easily assessed than yellows and reds, which is probably quite true, but of the eight colours examined six have blue as the predominating component, two have red, and yellows are conspicuous by their absence. This hardly seems a representative choice, and furthermore we are not informed whether the observers were trained or untrained.

It seems a pity, in a survey of this kind, that all the patterns examined should have been finished with melamine resin, particularly as the light exposures were made in a Fade-Ometer. It is well known that fading lamps of this type can give anomalous results with resin-finished materials.

Table II is somewhat surprising, as the observers apparently find it considerably more difficult to assess a pastel grey–blue than a pastel blue.

Finally, the conclusions drawn from this statistical analysis are most depressing. In assessing the fastness of one particular navy blue it is found that the accuracy is $\pm$ 1 unit and, if the writer's calculations are correct, with none of the remaining seven shades, except the turquoise, is it more precise. This presumably means that a light-fastness assessment figure may give the true fastness, or the pattern may be only half as fast, or it may be twice as fast. This would seem of doubtful value to the dye user, but does underline the wisdom of the words "without guarantee" appearing on dyemakers' pattern cards.

And what of those brave souls who have worked so long and so arduously to bring the assessment of light fastness to this state of imperfection? Will they echo the cry of old, "We have toiled all night and caught nothing?"

Whatever may be the value of making light-fastness assessments by the method used by the authors of this paper, it is very difficult to believe that assessment made by the present I.S.O. method of daylight exposures and with trained observers is of such doubtful accuracy.

G. G. Bradshaw

38 Altar Drive
Bradford 9
Yorkshire

14th November 1957

[1] Bülow, U., and Horrdin, S., J.S.D.C., **73**, 459 (1957).

It is evident that Mr. Bradshaw does not consider the uncertainty of the Swedish method SIS 650013 (= ASTM D 506–55) as representative also of the ISO method. We are, however, not convinced that the assessment variation of the ISO method is smaller than by SIS 650013. In both cases the same subjective decision must be made by the assessor (observer), viz. to decide whether the difference in colour of the fabric has reached a certain critical value. The latter then might be defined either in words such as "obvious", "just perceptible", etc. or by fixed standards, e.g. the grey scale. Of course, the process variation could not be lowered by the ISO method. Only the very insignificant testing variation *might* be depressed.

Without due cause maybe, we have assumed as being self-evident the fact that only persons with many years of experience of colour matching have made the assessments.

Regarding resin-treated fabrics, it is well known that the light fastness might be impaired by the treatment. As appears from our paper, the dyeing factor also may be affected. It is, however, not reasonable to assume that the assessment variation (time factor and observer factor) is affected.

The purpose of our investigation has by no means been "to bring the assessment of light fastness to a state of imperfection". That the investigation has given a somewhat depressing result about the certainty of the assessment is in our opinion not identical with the investigation being worthless. The value to the dye user is that he knows the import of the fastness grading and is aware of the actual uncertainty associated with the grading.

Finally, we regret that it is impossible for us to give a short lesson in statistical analysis in this limited space.

Ulf Bülow
Sture Horrdin

Borås Wäfveri AB.
Borås
Sweden

11th December 1957

Recognition of Chemical Damage in Wool

We enclose a reprint of our paper[1] entitled *Chemical Damage in Wool. Part I: Determination by Paper Chromatography*, which bears a close relationship to a paper, *The Use of Paper Chromatography in the Recognition of Chemical Damage to Wool* by V. Köpke and B. Nilssen, which appeared in the September 1957 issue of your *Journal*[2]. It is gratifying to see that others have found the same technique useful for determining chemical damage. We are, however, puzzled by the failure of your authors to note our earlier accomplishment as being similar to and quite as successful as their procedure. They do, of course, refer to our paper, but in a manner which does not suggest its content. In fact, their reference is to object that we overlooked the damaging of wool by certain uncommon alkaline materials which leave no lanthionine in the fibre. This is an objection which their method does not meet either, and will be true any time the damage is of the superficial surface type or where the damaged portion of the fibre is removed by the treatment.

Our principal point is to note the failure of your authors to extend the professional courtesy of referring to earlier, closely related research which, in fact, develops the main thought behind their work. We feel that your readers may be interested in knowing of the earlier work*.

W. H. Houff
R. H. Beaumont

F. C. Huyck & Sons
Rensselaer N.Y.
U.S.A.

15th October 1957

We are very grateful indeed for the comments by Mr. W. H. Houff and Mr. R. H. Beaumont both as regards the professional courtesy and the subject matter.

Obviously, our work was started long before their paper came to our hands, and as our results differed so much from Houff and Beaumont's, we thought it worth while publishing without further reference to their results, a thorough comparison being difficult as important details are missing from their paper. As an illustration, we mentioned the effect of alkali, which showed that sweeping statements like: "It has advantages over other methods in that it is specific for the detection of damage by these materials [i.e. alkalis and chlorine] and is not interfered with by other textile fibres" could hardly be very helpful. In this connection, we think that the recent work of Robinet and Bielen[3] on the effect of alkalis bears out our view that the chromatographic methods have serious limitations in this respect.

Vibeke Köpke
Bailli Nilssen

Norsk Tekstilforskningsinstitutt
Blindern
Norway

10th December 1957

[1] Houff, W. H., and Beaumont, R. H., *Text. Research J.*, **26**, 871 (1956).
[2] Köpke, V., and Nilssen, B., J.S.D.C., **73**, 413 (1957).
[3] Robinet, M., and Bielen, R., *Ann. Sci. Text. Belges*, (4), 31 (1956).

* An abstract of this paper appeared in the April 1957 issue of the *Journal— Editor.*

Lime-Zeolite Treatment of Water

Richardson[1] states that a slight excess of lime is added to the raw water in order to precipitate the temporary and magnesium hardness, the water then being passed through a bed of carbonaceous zeolite followed by a conventional base exchanger. The first stage— the treatment with lime— is carried out in the normal manner, the treated water then going forward to the carbonaceous zeolite.

The carbonaceous zeolite contains three active groups— (*a*) sulphonic, (*b*) carboxylic, and (*c*) phenolic— of which only the carboxylic groups are activated. This is achieved by adding the calculated amount of hydrochloric acid to the bed of carbonaceous zeolite and then rinsing through.

When a carbonaceous zeolite is being used in this manner, the sulphonic groups must be in the calcium form, as the zeolite is not stable at the pH of a lime-treated water (10·5) when the sulphonic groups are in the sodium form. Therefore, when a lime-treated water passes through the zeolite, the excess lime, calcium carbonate, and magnesium hydroxide in solution are all removed, with a consequent reduction in pH from 10·5 to 6·9–7·1. For obvious reasons, the permanent hardness is not altered and has to be removed by a conventional base-exchange treatment.

Thus, it is possible, by the above means, to treat a water containing bicarbonate hardness to give zero hardness and no alkalinity.

D. L. MORRALL

THE SPRINGFIELD HOSIERY FINISHING CO. LTD.
HUCKNALL LANE
BULWELL
NOTTINGHAM

28th November 1957

[1] Richardson, R. W., J.S.D.C., **75**, 485 (1957).

History of British Dyemaking

In connection with a history of the British dyemaking industry which I am writing, I am endeavouring to trace further information about the manufacturers of dyes and intermediates and the suppliers of raw materials to those firms. Many such firms are listed in the first edition of the *Colour Index* or are mentioned among the exhibitors at the International Exhibition of 1862 in Hofmann's Jurors' report. The great majority of these firms have long since ceased to exist. I should be grateful for any information either regarding the firms themselves or their activities or as to where information may be obtained or records consulted.

R. BRIGHTMAN

RED LODGE
COW LANE
NORLEY
CHESHIRE
VIA WARRINGTON

9th December 1957

ERRATUM

Feeder for Sodium Hydrosulphite. Anon. (J.S.D.C., **74**, 48 (Jan. 1958))— The reference should be *Amer. Dyestuff Rep.*, **46**, 499 (15 July 1957).

Notes

Meetings of Council and Committees

January

Council— No meeting

Finance and General Purposes— 28th

Diplomas— 15th

Publications— 21st

Textbooks and Monographs Subcommittee— 21st

Colour Index Editorial Panel— 10th

Historical Records— 31st

International Federation 1959 Congress Planning and Co-ordinating Committee— 21st

Deaths

We regret to report the loss by death of Mr. S. G. Corbishley and Mr. W. Poole.

A.S.D.C. Examination 1958

The annual examination for the Associateship of the Society of Dyers and Colourists will be held on Thursday, Friday, and Saturday, 29th, 30th, and 31st May 1958. Application forms are obtainable from the offices of the Society and must be completed and returned not later than 31st March 1958. Candidates should note that Papers A and B may be taken together, followed by C, D, and E; or papers A, B, and C may be taken together, followed by D and E.

D.S.I.R. Lending Library

The documents section of the Technical Information and Documents Unit (T.I.D.U.) has now been merged with the Lending Library Unit and operates from 20 Chester Terrace, Regents Park, London N.W.1.

All future requests for loan of documents referred to in T.I.D.U. Unpublished Report summaries should be addressed to the Lending Library Unit. Requests for photoreproductions of these reports, and of German documents, should also be sent there.

Loan copies of the published reports on German Industry in the BIOS, CIOS, and FIAT series are available from that address, and the German documents are also available for inspection between 9 a.m. and 5 p.m.

During the past six months the Unit has been collecting scientific and technological literature from the U.S.S.R., and believes that it now receives about half the current output.

Indanthren Dyes

German dyemaking firms have agreed that in future all new Indanthren dyes from Farbenfabriken Bayer AG. will have the prefix *E*, those from Farbwerke Hoechst AG. will have *H*, and those from Cassella Farbwerke Mainkur AG. *F*. Examples are Indanthren Brilliant Violet E5R powder superfine (FBy), Indanthren Blue HCBB (FH), and Indanthren Red FRC (CFM).

Rating of Scientific Societies

In Cane *versus* Society of Chemical Industry, the House of Lords has held, confirming a majority decision of the Court of Appeal, that the S.C.I. is exempt from rating by virtue of the Scientific Societies Act 1843.

Association Internationale de la Teinture Textile

At the annual congress of the A.I.T.T., held during June 1957 in Enschede in Holland, subjects discussed included problems of the European Common Market and Free Trade Area, modernisation of works, and price policy. It was reported that the A.I.T.T. was now recognised by the International Standards Organisation, and would thus be able to take part in the preparation of international test methods.

Problems of Scientific Education

The ninth Hinckley Memorial Lecture of the Institution of Chemical Engineers, *An Education for Our Times?*, was given by Dr. R. P. Linstead, F.R.S., Rector of the Imperial College of Science and Technology, on 5th November 1957. It is reproduced with only slight abridgment in *Chemistry and Industry* (1498–1502, 16th November 1957). Dr. Linstead emphasised the fact that, just as the Renaissance stimulated an interest in classical learning, so the special educational needs of today are scientific; but we have too few studying science and engineering, and among these the ratio of pure scientists to technologists is too high.

British higher education is among the shortest and most specialised in the world, but the lecturer recommended a careful and sympathetic approach to the problem of specialisation at school, aimed at securing greater flexibility in the general educational system. Although the nominal period for a first-degree course is three full-time years, the average at Imperial College is 3·3 years, because some students have to repeat years. At the Continental *Technische Hochschulen* the nominal period of study for a diploma is 4–5 years, but at one such institution the average time was in fact 7–8 years. Any immediate lengthening of the course for students of science and engineering in British universities would entail an additional capital sum of £15–30 million, a large increase in recurrent expenditure, and a hiatus in output for one year of some 7,000 graduates, and would thus be quite hopeless.

Ideas on the teaching of non-scientific subjects have not reached finality in either the Continental or the American technological institutions. In this country we should not introduce compulsory and examinable non-scientific subjects into the degree course, but should provide opportunities for the student to broaden his outlook.

Chair of Textile Chemistry in Aachen

The Director of the new German Wool Research Institute at Aachen, Prof. Dr.-Ing. Helmut Zahn, has been appointed as Professor Extraordinary to the new Chair of Textile Chemistry in the Rhenish–Westphalian Technische Hochschule in Aachen.

Dyes in Computers

According to *Industrial and Engineering Chemistry* (**49**, 23A (Aug. 1957)) the National Cash Register Corporation is carrying out preliminary work on the application of the principle of its "carbonless carbon paper" to the storage of information in computing machines. In the former, one side of each sheet of duplicating paper is coated with a film of capsules (2–3 μ. in diameter) of a material like gelatin containing a drop of oil coloured with a dye, and the other side bears a film of a claylike material; pressure from a pen or typewriter key breaks the capsules, and the dye reacts with the claylike coating on an adjacent sheet to produce the "carbon" copy. In the chemical memory device the dye would be photosensitive, and the transparent capsules would not be broken. Information would be stored as spots of a different colour produced by light of a suitable wavelength, and could be erased by light of another wavelength, which would return the spots to their original colour. The information could be "read" by light of a neutral wavelength, whose transmission or reflection would correspond to the colour pattern (stored information) on the surface; the effects could be amplified by suitable photomultiplier circuits, and the electronic signals transmitted to the computer.

Symposium on Chromatic Discrimination Paris, 25–29th July 1958

Although the symposium, on *Chromatic Discrimination in Animals and Man and the Problem of its Fundamental Mechanisms*, is being sponsored by the Psychological and Zoological Sections of the International Council of Scientific Unions, the tentative programme includes a preliminary session on methods and one or two sessions in which trichromatic and tetrachromatic physical schemes will be compared.

Participation in the symposium is by invitation, but some observers will be admitted on request to the Secretariat of the Symposium at the Collège de France, Place Marcelin-Berthelot, Paris V, France.

New Books and Publications

Fünf Kapitel Farbenlehre

By Eberhard Buchwald. Pp. 144. Mosbach/Baden: Physik Verlag. 1955. Price, DM 14.40.

Four of the five chapters of this book appeared in 1953–1954 in a scientific journal of the University of Jena, and the fifth, which deals with Goethe's work, is taken from a lecture by the author to the Goethe Society at Weimar in 1954.

Chapter I deals with the trichromatic system of colour specification including the C.I.E. system with three-dimensional colour space. The second chapter deals with "optimal" colours, which are those yielding maximum saturation for a given brightness. Additive and subtractive mixing are discussed in the section on colour printing and photography. The Ostwald system is discussed in

Chapter III, while Chapter IV returns to the trichromatic system with the plotting of colours in a rectangular uniform colour space.

A great deal of ground is covered in this small book, and for the person who is interested in quantitative colour measurement the treatment is rather abbreviated. On the other hand, 52 of the 116 pages are devoted to Ostwald and Goethe.

The references at the end of each chapter are predominantly German, and no recent references are quoted from journals printed in English.

While there is much of interest in this book, it is likely to be of limited appeal to those interested in colour in this country.

J. C. GUTHRIE

Farbenlehre und Farbenmessung

Eine kurze Einführung

By Werner Schultze. Pp. iv + 61. Berlin: Springer-Verlag. 1957. Price, DM 9.80.

This is a short introduction to the theory and measurement of colour, rather with a bias towards the theoretical considerations, although these are given in simple terms. Even the practical methods for the determination of colorimetric data are given in terms of the theory on which the methods are based without reference to specific types of instrument. However, references are given to other works in which both greater theoretical and practical detail are given.

The treatment is developed in a logical manner, separate consideration being given in the first instance to the visual mechanism, the nature of radiation (the spectrum), selective absorption and reflection of radiation by coloured substances, and also additive and subtractive colour mixing (by projection and Maxwell colour discs). Thus a simple but clear insight is given into the several main streams which flow together to form the theory of colour.

The C.I.E. system of colour specification is developed with clarity, but without reference to methods of manipulating colour equations. The method of representation in terms of chromaticity coordinates and luminance values is demonstrated by examples which add to the value of the explanation. The occurrence of visual matches (metameric) of coloured samples which give rise to different spectral energy distributions is rightly mentioned, as also is the non-uniformity of colour discrimination within the C.I.E. chromaticity diagram.

The colour systems developed by Ostwald and by Munsell are then introduced, and the more recent D.I.N. system (Manfred Richter) is explained. The essential connections between the Munsell and D.I.N. systems and C.I.E. coordinates are given, so as to allow a reasonable overall picture to be formed.

The experimental determination of colour is considered only in terms of the general principles of the three methods of approach using (*a*) a subtractive visual system, (*b*) spectrophotometric data, and (*c*) a direct photoelectric colorimeter. Here brief reference to specific instrumental systems in use would have added to the value of the information. The effect of fluorescent emission on selective reflection is obviously of some importance in these days, and it is pleasing to see a short account of this effect and its measurement.

The practical application of colour measurements is finally considered with reference to several different fields, and completes a work which gives a reasonable introduction to colour and its measurement.

ERIC COATES

Colour in Surface Coatings

Its Appreciation, Measurement, and Control

The Research Association of British Paint, Colour, and Varnish Manufacturers. Pp. 68. Teddington: the Association. 1956. Price, 7s. 6d.

This is a brief account of the properties of radiation (spectral energy distribution, interaction with matter, stimulation of the eye, and photoelectric devices) in so far as these are related to the appreciation, measurement, and control of colour. An attempt has been made to present to those who are interested in colour, but who have no great technical knowledge of the subject, a simple picture of the complex process of seeing colour. Indeed, the sparsity of technical terms and definitions is remarkable. This could, perhaps, be misleading in some instances (e.g. no distinction is made between saturation and Munsell chroma), but on the whole the brochure should provide understandable and interesting reading to those for whom it is intended. The lack of references to more detailed works will no doubt be disturbing to those who become deeply interested in the subject-matter, although the necessary physiological, psychological, and physical considerations are outlined to make the book fairly well self-contained if no more than an appreciation of the elements of the subject is required.

The few examples of surface colours given and the reference to colour systems for classification are considered from the point of view of the paint manufacturer, but this does not detract from the main part of the text for anyone interested in surface colours of any other type.

The industrial applications of colour measurements are becoming of increasing importance and interest, and are discussed fairly and adequately, in relation to the rest of the text. The assistance which colour measurements could give to the colour matcher is emphasised, and yet it is properly acknowledged that the experienced eye is more than a match for the photocell, at least for the present.

ERIC COATES

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

Safety in the Chemical Industry

American Chemical Society

Ind. Eng. Chem., **49**, 1727–1754 (Oct. 1957)

Symposium of seven papers dealing with— integration of safety principles into a development programme, laboratory safety programme for hazardous catalysts and catalyst initiators, venting dust explosions, monitoring of solvent–air mixtures with infrared analysers, barricading hazardous reactions, standards for laboratory sample cylinders, and safe practices for handling compressed gases.

C.J.W.H.

Mechanisation of Ancillary Operations in Finishing

A. I. Dergacheva

Tekstil. prom., **17**, 44–47 (June 1957)

A number of mechanical ancillary operations (e.g. feeding of damp material on a conveyor belt system) already in use in finishing and printing works are described, and a much wider use is recommended. G.J.K.

PATENTS

Treating Textiles with Corrosive Gases and/or Vapours

J. Dungler *BP* 783,287

When chlorite bleaching by the steam process, corrosion of the apparatus is avoided if the impregnated material is passed through a chamber in which the impregnating liquor is converted into a superheated vapour, while extracting any excess vapour to avoid saturation pressure being reached in the chamber. C.O.C.

Cone for use in the Package Dyeing of Yarn

Russell Manufacturing Co. *USP* 2,746,280

A cone of moulded plastic has a central bore to fit it on a package dyeing machine spindle and a winder spindle adapter. The wall has perforations in it to allow the dye liquor to pass through. This wall increases in thickness towards the large end of the cone to give increasing strength to withstand increased shrinkage stresses imposed towards this large end by a package of yarn supported on the cone. C.O.C.

Adjusting Squeeze Rollers

Sellers & Co. (Huddersfield) *BP* 784,728

Modification of *BP* 629,310 (J.S.D.C., **66**, 151 (1950)) pneumatic means being used instead of hydraulic means to adjust the squeeze between a pair of rollers. C.O.C.

High-Speed Roller Coating Machine

Metal Box Co. *BP* 772,182

Patchy coating at high operating speeds of roller coating machines is obviated by supplying the lacquer feed as a spray uniformly distributed along the nip between the feed roll and the transfer roll. Alternatively the feed roll may be replaced by a fixed doctor blade. E.C.

Boarding and Presetting Apparatus for Hosiery

Sarco *USP* 2,745,191

Applying Hosiery Transfers

Paramount Textile Machinery Co. *BP* 782,002

Transfers are applied automatically and continuously to hose with means for altering the relative positions of transferring points to accommodate differences in size and shape of hose. G.E.K.

Cloth Folding Machines having an Automatic Edge Guide

Lever Bias Machine Corpn. *BP* 784,878

II—WATER AND EFFLUENTS

Supply, Treatment, and Disposal of Water in the Dyehouse

R. W. Richardson

J.S.D.C., **73**, 485–491 (Nov. 1957)

An attempt is made to review briefly the many aspects of water— its supply, treatment, and disposal— from the point of view of dyehouse management and the dyer, rather than from that of the water-treatment specialist.

AUTHOR

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Annual Review of the Literature on Fats, Oils, and Detergents

M. M. Piskur

J. Amer. Oil Chem. Soc., **34**, 222–239 (May), 264–293 (June 1957)

Statistical-mechanical Theory of Micelle Formation in Detergent Solutions

C. A. J. Hoeve and G. C. Benson

J. Physical Chem., **61**, 1149–1158 (Sept. 1957)

A general theory for micellar solutions is developed. Possible distributions of micellar size are derived for non-ionic detergents. When large micelles are formed, the predicted shape is plate-like rather than rod-like.

W.R.M.

Mothproofing Agents for Wool. XXV— Synthesis of Chloromethanesulphonamides— Constitution of Eulan WA (FBy)

K. Matsui, H. Kikuchi, and I. Miyazaki

Yûki Gôsei Kagaku Kyôkaishi, **15**, 395–399 (1957): *Chem. Abs.*, **51**, 16325 (20 Nov. 1957)

Evidence which shows that the constitution of Eulan WA (FBy) is $ClCH_2SO_2NHC_6H_2Cl_3$–2:4:5. C.O.C.

PATENTS

Werner Complexes as Antistatic Agents

DuP *USP* 2,745,770

Substances having regain $> 5\%$ and a resistivity of $< 400{,}000 \times 10^6$ ohms are treated with a Werner complex in which a trivalent Cr atom is coordinated with a *p*-nitrophenylaceto, *p*-nitrobenzoaceto, phenylaceto, trichloroaceto, thioglycollato, cyanoacetato or gluconato group.

C.O.C.

Detergent

Shell Refining & Marketing Co. *BP* 784,228

Mixtures of liquid synthetic detergents and soaps are more stable at low temperatures, e.g. 10°C., if the soaps are ammonium and not sodium or potassium salts.

C.O.C.

Sodium Metasilicate Compositions containing Fluorescent Brightening Agents

Alcock (Peroxide) *BP* 784,538

A fluorescent brightening agent is added to a melt of sodium metasilicate which is then allowed to crystallise. The product is suitable for washing cellulosic textiles.

C.O.C.

Bleaching Composition

Colgate-Palmolive Co. *BP* 784,452

A powdered bleaching composition contains 1–30% (by wt. of the total composition) 1:3-dichlorodialkylhydantoin, preferably 1:3-dichloro-5:5-dimethylhydantoin, and $< 10\%$ of a molecularly dehydrated phosphate, alkali metal silicate or alkali metal carbonate. The remainder of the material is made up of alkali metal salts (to bring the total alkali metal salts to 60–90%) and surface active agents. C.O.C.

Cationic Softening Agents for Cellulose

Ciba *BP* 782,974

The water-soluble quaternary ammonium compounds obtained by heating an aralkyl halide or an alkyl halide containing a carboxylic acid amide group or an epoxy group with an aliphatic or *cyclo*aliphatic tertiary amine containing > 1 aliphatic or cycloaliphatic residue of > 7 C, have cation active properties and strong affinity for cellulose. They are used as auxiliary agents and particularly as softening agents. The amine must be the condensate of an *N*-methylol derivative of an aliphatic or *cyclo*aliphatic carboxylic acid amide or urethane of > 7 C with an *N*-hydroxyalkyl aliphatic or *cyclo*aliphatic tertiary amine (hydroxyalkyl of > 1 C). Thus the condensate of stearic acid-*N*-methylolamide and triethanolamino is heated with benzyl chloride to yield a light coloured product of use as a softening agent for viscose rayon. C.O.C.

Amphoteric Softening Agents Stable to High Temperatures and Hard Water
BASF *BP* 784,669

Condensing high mol.wt. fatty acids with trihydroxyalkyl dialkylene triamines and then treating with dibasic carboxylic acids to yield a product containing some unreacted carboxyl groups yields softening agents which are soluble both in acidic and alkaline solutions, inert to hard water and which impart a soft handle unaffected by drying or other high temperatures to which the treated goods are subjected during processing. C.O.C.

Dicarbamate-formaldehyde Resins— Textile Finishes
Rohm & Haas Co. *BP* 774,749

Water-soluble condensation products are prepared from diethylene glycol dicarbamate (1 mole) and aqueous formaldehyde (2–3·5 mole) at 100°c. approx., finally at pH 3 until the viscosity of the aqueous solution at 60% solids content is 2–25 poise at 25°c. The products are useful for treating paper and textiles and after heating give moisture and grease-resistant coatings. E.C.

Stable Resinous Precondensates as Textile Finishes
Boehme Fettchemie *BP* 783,123

Precondensates of excellent stability and which can readily be condensed with acid-adjusted water-repellent agents are obtained by treating an organic nitrogen compound containing amino and/or imino-groups with >1 mol. of an aldehyde to each amino- or imino-group. Thus, urea (6 Kg.) is dissolved in 30% formalin (24 litres) at pH 7·2 and heated at 80°c. for 20 min. and cooled. This yields a soft paste of good resistance to ageing. This paste is dissolved in water and mixed with an acidic emulsion of paraffin wax containing a zirconium salt. The resulting liquor is stable for at least 24 hr. It is used to confer both ability to recover from creasing and water-repellent properties to spun rayon fabrics. C.O.C.

Nitrogen-containing Polyvinyl Ethers— Textile Finishes
Rohm & Haas Co. *BP* 772,666

N-Vinyloxyalkyl alkylenediamines (alkyl and alkylene $>C_1$) are heated with urea to form an imidazolone or a hexahydropyrimidone ring. The vinyl ethers thus obtained polymerise readily with free radical catalysts and are useful as modifiers for urea and melamine resins. The vinyl ether polymers are useful as warp sizes, textile finishes, adhesives and coatings and their methylol derivatives (from formaldehyde) are useful in treating paper and leather and for stabilising fabrics of wool and rayon. E.C.

Non-inflammable Heat-convertible Copolymers
Solvay & Cie. *BP* 772,978

Copolymers of vinyl or vinylidene chloride (80–99%) and allyl acrylate, methacrylate or α-chloroacrylate (1–20%) are prepared in solution, e.g. in acetone, and are soluble, fusible and non-inflammable. On heating alone or with a polymerisation catalyst they become insoluble and infusible. The fusible polymers are useful for impregnating textiles and glass fabrics, to produce laminates, and for surface coatings, e.g. on leatheroloth. E.C.

Dextran Xanthate as the Adhesive in making Non-woven Fabrics
Commonwealth Engineering Co. of Ohio *BP* 784,671

Dextran xanthate is a cheap and efficient adhesive for use in making non-woven fabrics. C.O.C.

Alkali-soluble Bisphenol Resins
S. C. Johnson & Son *BP* 772,301

Alkali-soluble resins are prepared by condensing under aqueous alkaline conditions a bisphenol, e.g. diphenylolpropane with, firstly, an epoxyhalide, aliphatic dihalide or diepoxide or dihalo-ether, e.g. glycerol epichlorohydrin, and secondly a monoepoxy or monohalo-acid, e.g. chloracetic acid. They are useful in making paints and fabric-treating compositions or may be dissolved in aqueous ammonia and added to conventional self-polishing wax or shellac emulsion polishes to give spot-free washable coatings of improved gloss and durability. E.C.

Chloroacetic Esters as Tanning Agents
Henkel & Cie *USP* 2,746,838

Chloroacetic acid esters of fatty alcohols are very good tanning agents for any kind of skin or hide. C.O.C.

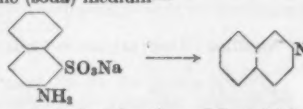
IV— RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Mechanism of the Isomerisation of α- into β-Naphthalenesulphonic Acid
F. M. Vainshtein and E. A. Shilov
J. Gen. Chem. U.S.S.R., **27**, 2559–2566 (Sept. 1957)

Naphthalene Derivatives

XV— Rearrangements of Aromatic Sulphonates; Rearrangement of the Dry Salts of 2-Naphthylamine-1-sulphonic Acid and 2-Naphthylsulphamic Acid
V. V. Kozlov
J. Gen. Chem. U.S.S.R., **27**, 1146–1155 (May 1957)

Heating (at 100–170°c.) the dry sodium salt of 2-naphthylamine-1-sulphonic acid (I) causes rearrangement to the salt of β-naphthylsulphamic acid, which is stable in alkaline (soda) medium—



Prolonged (12 hr.) heating of I at higher temp. (218°c.) leads to the formation of the salt of 2-naphthylamine-6-sulphonic acid.

XVI— Rearrangement of 2-Naphthol-6-sulphonate to 2-Naphthol-7-sulphonate
V. V. Kozlov and G. G. Yakobson
Ibid., 1156–1160

"Baking" the dry sodium salt of 2-naphthol-6-sulphonic acid (8 hr. at 330°c.) leads to the formation of a 2-naphthol-7-sulphonate (ca. 50% yield). G.J.K.

Interaction of Keten with Nitrogen-containing Bases. III— Acetylation of Primary Aromatic Amines with Keten
Yu. V. Svetkin
J. Gen. Chem. U.S.S.R., **27**, 1676–1679 (June 1957)

Acetylation involving the use of keten of *o*- and *p*-aminophenol and *o*- and *p*-nitroaniline is investigated. Hydrogen bonding in the latter compounds is held responsible for the difficulty of interaction. Acetylation of many other aromatic amines (at temp. of 20–40°c.) produces an almost quantitative yield of the *N*-acetyl derivative. The first preparation of *N*-acetyl-4-nitro-2-aminophenol is recorded. G.J.K.

Molecular Polarisation and Molecular Interaction. VII— Apparent Dipole Moments of some Di- and Tri-substituted Anilines in Benzene and Dioxan
J. W. Smith and S. M. Walshaw
J.C.S., 4527–4531 (Nov. 1957)

The apparent dipole moments of 2:4- and 3:5-dibromoaniline, 2:4- and 3:5-dinitroaniline, 2:4:6-trinitroaniline, and 2-methyl-4-nitroaniline in benzene and dioxan are recorded, and the interaction moments μ_{int} and the vectorial values of the additional moments in dioxan $\Delta\mu_{vect}$ for these and allied amines are calculated. For the disubstituted anilines, μ_{int} and $\Delta\mu_{vect}$ follow the linear relation observed for *m*- and *p*-monosubstituted anilines. The values of μ_{int} for 2:4:6-tribromo- and 2:4:6-trinitroaniline indicate little or no steric inhibition of the mesomeric effect of the amino group such as is observed with mesidine and aminodurene. For all aniline deriv. containing two *ortho* groups, however, $\Delta\mu_{vect}$ is less than would be expected from the linear relationship. The results indicate that, if not more than one position *ortho* to the amino group is occupied by a substituent, the difference between the apparent dipole moments of an amine in dioxan and benzene soln. serves as a reliable guide to the extent of the mesomeric effect of the amino group in the free amine. H.H.H.

Thermal Analysis of Binary Systems of Benzidine with Phenols and Naphthols
A. G. Bergman and A. P. Arestenko
J. Gen. Chem. U.S.S.R., **27**, 867–870 (April 1957)

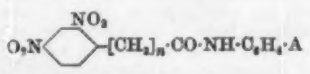
Binary systems comprising benzidine with mono- and dihydroxybenzenes and with naphthols are investigated by thermal analysis, phase diagrams being given. The introduction of a second hydroxyl group in benzene facilitates the formation of molecular complexes, whereas a nitro group *ortho* to the hydroxyl has the opposite effect. G.J.K.

Ternary System Benzidine–Phenol–Naphthalene
A. G. Bergman, A. P. Arestenko, and A. I. Kislova
J. Gen. Chem. U.S.S.R., **27**, 870–875 (April 1957)
This ternary system has been investigated by thermal analysis. Formation of the complex $(C_6H_4)_2(NH_2)_2$, $2C_6H_5OH$ is greatest at 141°C. (75·33% of the triangular area). G.J.K.

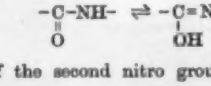
Spectrophotometric Study of Diphenylamine and its Derivatives in Concentrated Sulphuric Acid—I
P. M. Bugaĭ
J. Gen. Chem. U.S.S.R., **27**, 1632–1641 (June 1957)
Solution in conc. H_2SO_4 of diphenylamine and its 4-hydroxy and 4-methoxy derivatives produces deeply coloured solutions, the colour of which does not change on great dilution. G.J.K.

Nitration of easily Nitratable Aromatic Compounds
R. A. Slavinskaya
J. Gen. Chem. U.S.S.R., **27**, 1160–1167 (May 1957)
The author investigates the nitration of phenol, *o*-nitrophenol, *p*-nitrophenol, phenetole, naphthalene, and acetanilide with nitric acid in ethyl nitrate at 18°C. The nitration of phenol leads to the formation of *o*-, and *p*-nitrophenol and 2:4-dinitrophenol, *p*-nitrophenol undergoing nitration to dinitrophenol much more readily than the *ortho* isomer. Phenetole and napthalene yield mononitro derivatives, whilst acetanilide does not undergo nitration under these conditions. Nitration is attributed to the presence of NO_2 in nitric acid. G.J.K.

Colour of Arylamides of 2:4-Dinitrobenzoic Acid
E. A. Smirnov
J. Gen. Chem. U.S.S.R., **27**, 1922–1933 (July 1957)
The system—



(A = O·CH_2, OH, or $N(CH_3)_2$ *meta* or *para* to the NH group) is studied by means of reflectance and absorption spectra. These compounds are deeply coloured, and the explanation in the case of the arylamides of dinitrobenzoic and dinitrocinnamic acids is in terms of a tautomeric conjugated system—



The effect of the second nitro group upon spectra is mentioned. G.J.K.

Study of Tautomeric Systems by means of Labelled Atoms. I— Triazen (Diazoamino) System
M. M. Shemyakin, V. I. Maĭmind, and E. Gomes
J. Gen. Chem. U.S.S.R., **27**, 1842–1849 (July 1957)
The preparation of diazoamino systems containing one ^{15}N atom is given. These compounds are used to investigate the tautomerism possible within this system. The authors claim that they have shown for the first time the existence of two tautomeric forms, e.g.—

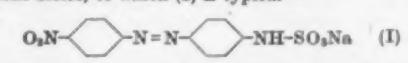
$$C_6H_5{-}^{14}N = {}^{14}N{-}^{15}NH{-}C_6H_5 \quad (I)$$
$$C_6H_5{-}^{14}NH{-}^{14}N{-}^{15}N{-}C_6H_5 \quad (II)$$

and they emphasise the mobility of such a system. G.J.K.

Fluorine-containing Derivatives of Dimethylaminoazobenzene
L. M. Yagupol'skiĭ and M. S. Marenets
J. Gen. Chem. U.S.S.R., **27**, 1395–1399 (May 1957)
The synthesis of a series of fluorine-containing dimethylaminoazobenzenes is carried out and their spectra in acid and alkaline solution are determined. Introduction of fluorine causes a hypsochromic displacement of the absorption band. G.J.K.

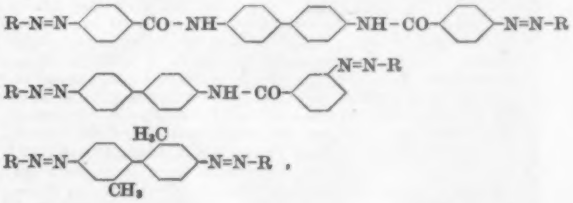
Reaction of Diazo Compounds with Sulphamic Acid and its Derivatives
D. Z. Zavel'skiĭ and L. A. Lishnevskaya
V— Reactions of Diazo Compounds with *N*-Phenylsulphamic Acid
J. Gen. Chem. U.S.S.R., **27**, 1330–1339 (May 1957)
At low pH values diazo compounds and phenylsulphamic acid form the diazonium phenylsulphamates $Ar{\cdot}N_2{\cdot}O{\cdot}SO_2{\cdot}NH{\cdot}C_6H_5$, which are deeply coloured in aqueous solution. Between pH 4 and 6 they undergo rearrangement mainly to the unstable diaryltriazen-*N*-sulphonic acids $Ar{\cdot}N{:}N{\cdot}N(SO_2Na){\cdot}C_6H_5$ with subsequent decomposition to (the asymmetric) diaryltriazen and sulphuric acid. Compounds of the type $Ar{\cdot}N{:}N{\cdot}C_6H_4{\cdot}NH{\cdot}SO_2Na$, with coupling occurring in the *para* position, are obtained in a side-reaction.

VI— Indicator Properties of 4-Aminoazobenzene-*N*-sulphonates
Ibid., 1339–1345
Absorption spectra of dyes of the *N*-sulpho-4-aminoazobenzene series, of which (I) is typical—



are given. Compounds of this type are indicators giving colour changes in both acid and alkaline medium. Their spectra are explained on the basis of modern electronic theory. G.J.K.

Effect of Steric Factors on the Properties of Dyes containing the Diphenyl System.
VIII— Bisazo Dyes from *m*- and *p*-Aminobenzoyl Derivatives of Benzidine and 2:2′-Dimethylbenzidine
B. M. Krasovitskiĭ, B. I. Ostrovskaya, and V. B. Smelyakova
Ukrainian Chem. J., **23**, 496–500 (Aug. 1957)
The preparation and analysis of a series of bisazo dyes, e.g.—



are described, and the absorption spectra in aqueous solution determined. The effect of the position of substituents upon the colour and the substantivity towards cellulose is discussed.

IX— Effect on the Colour of Monoazo Dyes derived from Diphenyl, Fluorene, and Dinaphthyl
B. M. Krasovitskiĭ, L. M. Litvinenko, and T. A. Serova
Ibid., 501–504
The absorption spectra in aqueous solution of a series of monoazo dyes derived from benzene, diphenyl, fluorene, and dinaphthyl are determined, and the effect of a substituent upon the coplanarity of the benzene rings of the "effective" system is correlated with the colour of the dye. G.J.K.

Diamagnetic Susceptibilities of Azo Derivatives of 2-Naphthol
Y. Matsunaga
Bull. Chem. Soc. Japan, **30**, 429–430 (June 1957)
Magnetic susceptibility measurements on solid phenylazo-2-naphthols substituted in the benzene ring show that the *o*-CH_3, *m*-NO_2, *p*-NO_2, and unsubstituted compounds exist in the quinone–hydrazone form, and the *p*-CH_3, *o*-CH_3O, *o*-Cl, and *p*-Cl derivatives in the azophenol form. These results are at variance with those of Burawoy *et al.* (*J.C.S.*, 4793 (1952)) for phenylazo-2-naphthols in soln. A.J.

Relative Stabilities of Metal Derivatives of *oo′*-Dihydroxyazo Dyes
F. A. Snavely, W. C. Fernelius, and B. E. Douglas
J.S.D.C., **73**, 491–495 (Nov. 1957)
The relative stabilities of metal derivatives of *oo′*-dihydroxyazo dyes (terdentate groups) have been measured potentiometrically in dioxan–water (75 : 25 by volume). The order of stability of the metal derivatives agrees with that already reported in the literature for monodentate and bidentate groups. The coordinating power of the diarylazo compounds for a given metal is proportional to the acid dissociation constants of the chelating agents. The one *o*-hydroxy-*o′*-carboxyazo dye which has been studied forms less stable metal derivatives than the *oo′*-dihydroxyazo dyes and differs from them in coordinating with beryllium. AUTHORS

Crystal Structure of a Monoclinic Form of Naphthazarin
H. Watase, K. Osaki, and I. Nitta
Bull. Chem. Soc. Japan, **30**, 532–536 (July 1957)

Products of Condensation of Arylcarbinols with 1-Phenyl-3-methyl-5-pyrazolone— III
O. F. Ginzburg, Ts. M. Gol'dberg, and É. I. Kvyat
J. Gen. Chem. U.S.S.R., **27**, 993–997 (April 1957)
The condensation of 9-phenylxanthhydrol with 1-phenyl-3-methyl-5-pyrazolone (I) takes place in alcohol and also in acetic acid. These condensation products of arylcarbinols with I are decomposed in alkaline aqueous–alcoholic solution. G.J.K.

Indicator Colour Changes
O. F. Ginzburg and B. A. Poraĭ-Koshits
J. Gen. Chem. U.S.S.R., **27**, 989–992 (April 1957)
An investigation into the indicator properties of compounds such as *p*-nitrophenol and Malachite Green (C.I. Basic Green 4) confirms Ostwald's conclusions concerning the nature of indicators. If an indicator exists in two tautomeric forms, then the "apparent" ionisation constant characterises the less ionised form. Not only the ionisation constant but also the rate at which equilibrium is reached are important for the choice of a suitable indicator. G.J.K.

State of Redox Indicators in Solution
B. P. Nikol'skiĭ and V. V. Pal'chevskiĭ
Izvestiya Akad. Nauk S.S.S.R. otdel khim. nauk, (5), 532–540 (May 1957)
Replacement of the calomel electrode, used in Clark's method (*Chem. Rev.*, **2**, 127 (1925)) for the potentiometric determination of dissociation constants of redox systems in solution, by the hydrogen electrode (without transport in the system) allows the e.m.f.–pH relationship to be used for more accurate determinations of dissociation constants. With the aid of graphs, the redox systems of Methylene Blue, Toluidine Blue, and Thionine (C.I. Basic Blues 9, 17, and 25) are evaluated; changes in ionic strength from 0·013 to 0·8 do not affect the e.m.f. The results of these potentiometric investigations are found to be in good agreement with those obtained independently by the spectrophotometric technique. G.J.K.

Free Radicals and Radical Stability

XIII— Triphenylmethyls containing *m*-Methoxyl Substituents
S. T. Bowden
J.C.S., 4235–4239 (Oct. 1957)

XIV— (Ethoxyphenyl)diphenylmethanols and the Free Radical (*o*-Ethoxyphenyl)diphenylmethyl
S. T. Bowden and D. T. Zalichi
Ibid., 4240–4243

XV— Properties of (*o*-Hydroxyphenyl)diphenylmethanol and (3-Hydroxy-2-naphthyl)diphenylmethanol
S. T. Bowden and K. I. Beynon
Ibid., 4244–4247
The colour developed on heating soln. of (*o*-hydroxyphenyl)- and (3-hydroxy-2-naphthyl)-diphenylmethanol is shown, from studies of the absorption spectra of hot soln. in acetic acid, to be a type of thermochromism arising from the broadening of a near-ultraviolet absorption band and not due to formation of the carbonium ion. Decomp. of the alcohols at elevated temp. is explained in terms of a free-radical mechanism which does not involve formation of quinonoid intermediates. The alcohols give rise to carbonium ions in H_2SO_4, and the presence of the *o*-hydroxyl group in the triphenylmethyl and 2-naphthyldiphenylmethyl ions leads to a bathochromic shift in the absorption spectrum, and also promotes sulphonation in H_2SO_4.

XVI— Properties of Triarylmethanols containing *p*-Hydroxyl Substituents, and the Quinonoidation Theory of Free Radicals
K. I. Beynon and S. T. Bowden
Ibid., 4247–4253
Diphenylquinomethane and the so called colourless and coloured forms of (*p*-hydroxyphenyl)diphenylmethanol give the same carbonium ion in H_2SO_4, and their hot formic-acid soln. evolve CO_2 at practically the same rate. The present analysis of the absorption spectra of mixtures of colourless alcohol and diphenylquinomethane, and the X-ray and thaw-melt examination of the solid systems, indicate the colour to be due to the presence of the fuchsone. The properties of other triarylmethanols containing *p*-hydroxyl substituents may be accounted for on a similar basis. If, as indicated, quinonoid forms of these cpd. have no independent existence, Gomberg's main evidence in support of the quinonoidation theory of free radicals is no longer valid.

XVII— (*m*-Hydroxyphenyl)diphenylmethanol and the Corresponding Free Radical
S. T. Bowden and K. I. Beynon
Ibid., 4253–4256
(*m*-Hydroxyphenyl)diphenylmethanol does not exhibit thermochromism or form a fuchsone, and the carbonium ion is less readily sulphonated than the *o*-substituted ion. (*m*-Hydroxyphenyl)diphenylmethyl chloride is reduced by silver to the bright yellow free radical, and the corresponding di-(*m*-hydroxyphenyl)tetraphenylethane is the first deriv. of this type to be isolated in the solid state. Although the presence of the hydroxyl group in the radical may lead to the formation of hydrogen-bonded complexes, the observed unimol. stability is greater than that of (*m*-methoxyphenyl)diphenylmethyl owing to the stronger inductive effect of the substituent.

XVIII— (Halogenophenyl)diphenylmethyls and the Removal of Nuclear Halogen by Silver
K. I. Beynon and S. T. Bowden
Ibid., 4257–4262
In contrast to *para* substitution, *meta* substitution by halogen in the triphenylmethyl cation leads to a slight hypsochromic shift in the absorption spectrum. (*m*-Halogenophenyl)diphenylmethyl chlorides do not form fuchsones, and are reduced to primary radicals which may be isolated in the solid state as ethanes. Silver removes half of the halogen from (*p*-bromophenyl)diphenylmethyl, but has no action on a radical with *meta* halogen; this behaviour is interpreted without assuming the existence of a *para*-quinonoid form of the radical (cf. *J. Amer. Chem. Soc.*, **36**, 1157 (1914)). The interpretation also accounts for the differences in the reactivity of *para*- and *meta*-substituted radicals towards silver, and for the apparently anomalous behaviour of (*p*-fluorophenyl)diphenylmethyl, which cannot be explained on the basis of the Gomberg theory. H.H.H.

Benzidine-type Rearrangement of Aromatic Disulphides
H. J. Shine and J. L. Bear
Chem. and Ind., 565–566 (4 May 1957)
The reported work confirms that a benzidine-type rearrangement takes place with diphenyl disulphide on treatment with 100% H_2SO_4. When the acid is $< 100\%$ a series of partly oxidised or sulphonated polymeric disulphides of the form—

$$-(S{\cdot}C_6H_4{\cdot}C_6H_4{\cdot}S{\cdot}S{\cdot}C_6H_4{\cdot}C_6H_4{\cdot}S)-$$

obtain. This may account for the failure of Szmant and Lapinski (*J. Org. Chem.*, **21**, 847 (1956)) to confirm the findings of the authors and others, that such a rearrangement does in fact occur. J.W.D.

Cyanine Dyes

VII— Cyclisation of Secondary Aromatic Amines with Vinyl Ethers to give Arylquinaldines
G. T. Pilyugin and E. P. Opanasenko
J. Gen. Chem. U.S.S.R., **27**, 1015–1018 (April 1957)
Interaction of secondary aromatic amines with vinyl butyl and vinyl ethyl ethers in acid medium causes ring closure with the formation of *N*-arylquinaldines. The quaternary ammonium perchlorate and iodides of such compounds are given, the yields in this instance being greater than those obtained from paraldehyde.

VIII— Synthesis and Reactions of the Alkoxy Derivatives of Quaternary Salts of Benzoquinaldine
G. T. Pilyugin, E. P. Opanasenko, and N. A. Tsvetkova
Ibid., 1018–1021
The interaction of paraldehyde with *p*-methoxyphenyl-β-naphthylamine and with *p*-ethoxyphenyl-α-naphthylamine to give *N*-arylquinaldines is studied, and some new quaternary salts are characterised. Carbocyanine and *iso*-cyanine derivatives are obtained from these salts. G.J.K.

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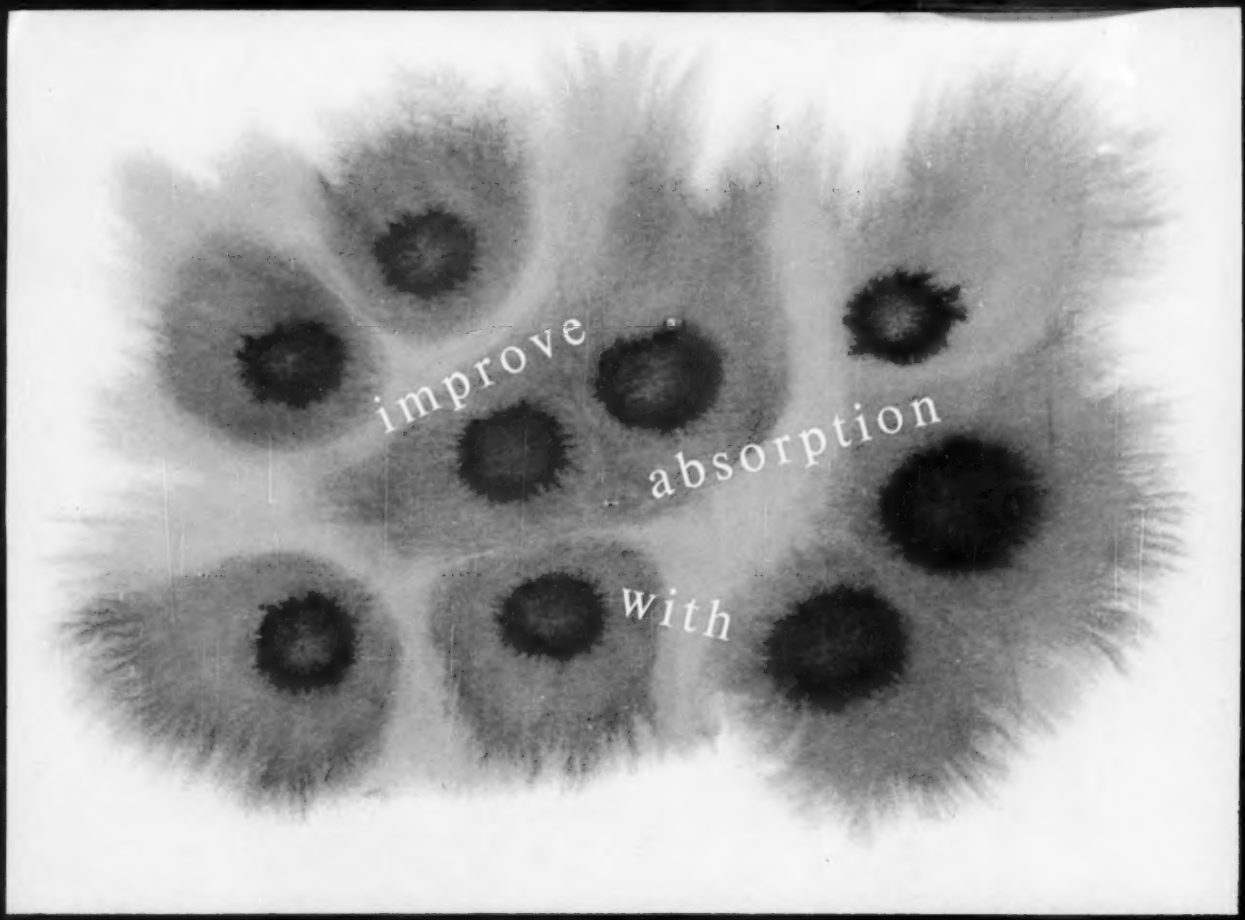

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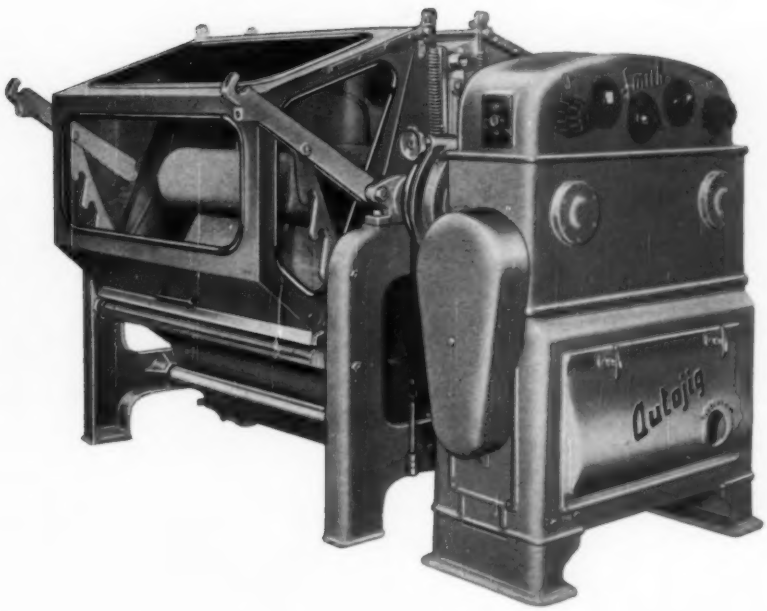

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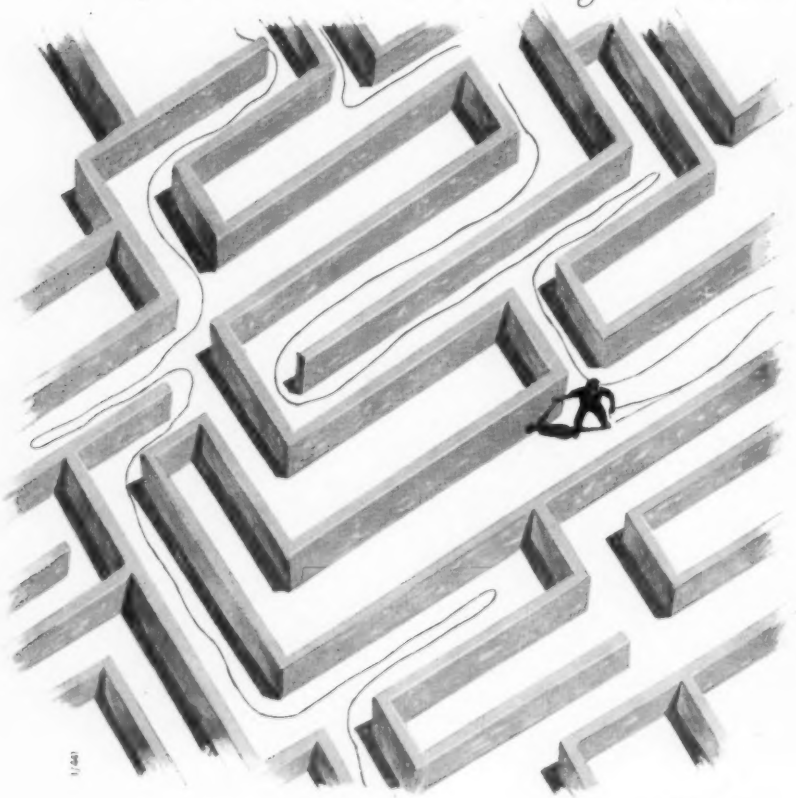

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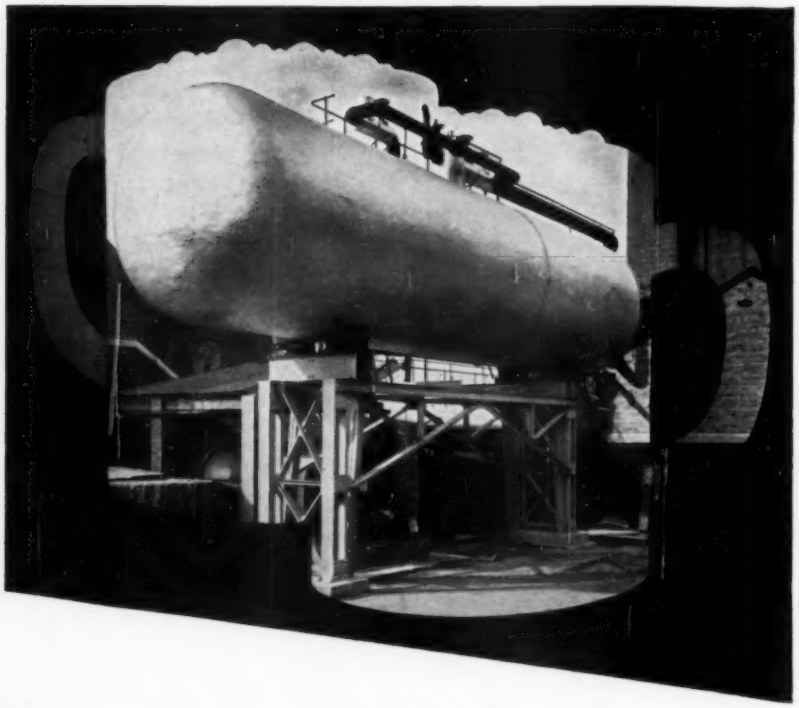

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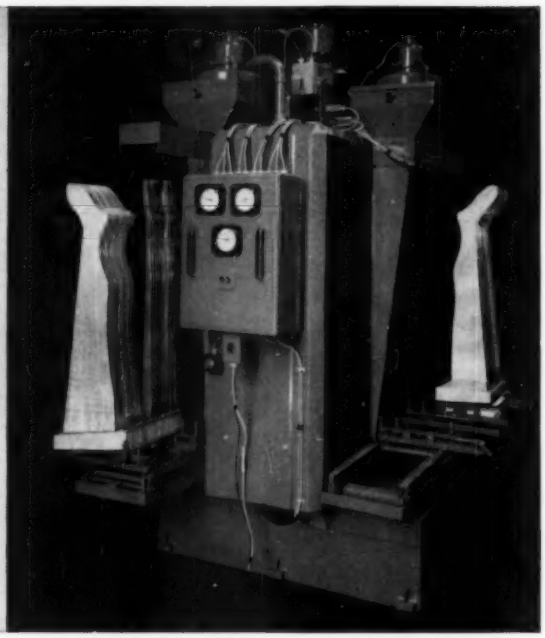

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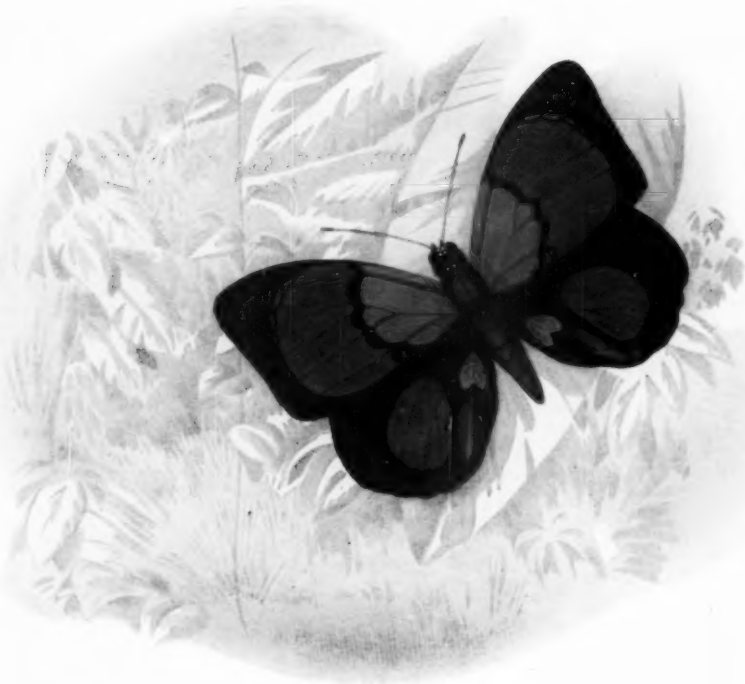

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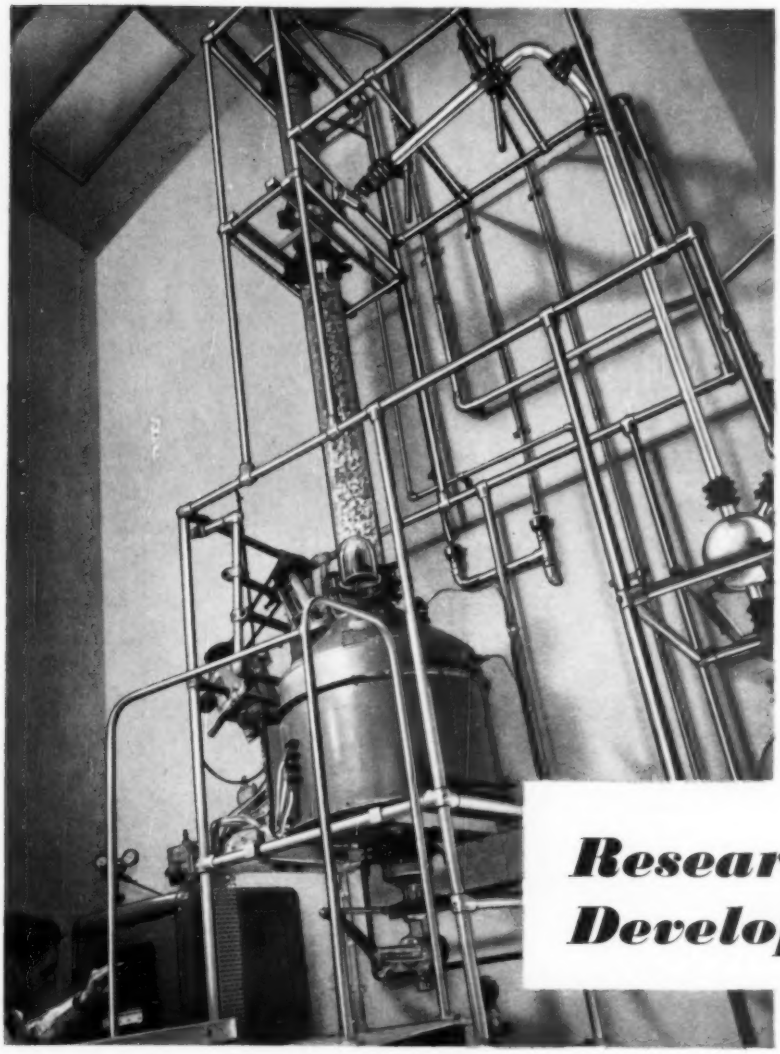

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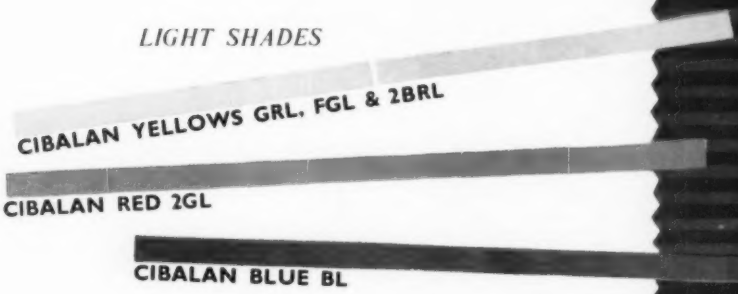

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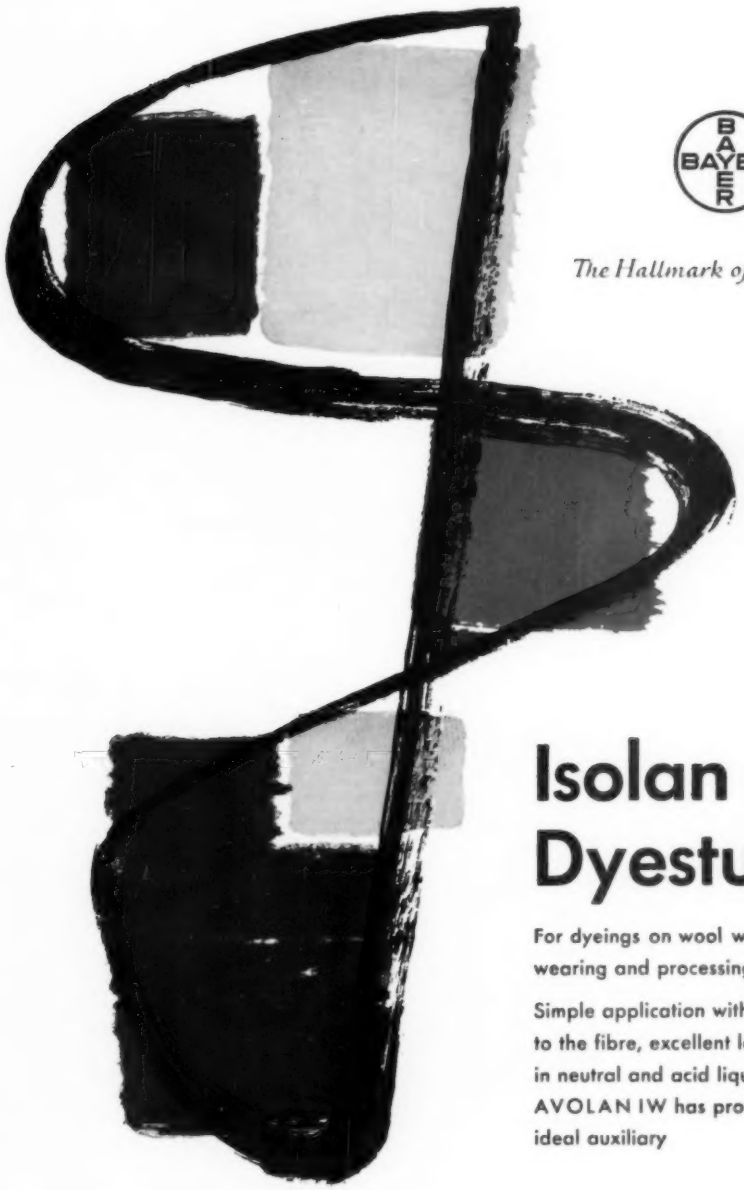

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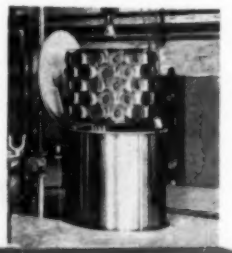

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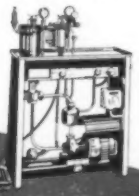

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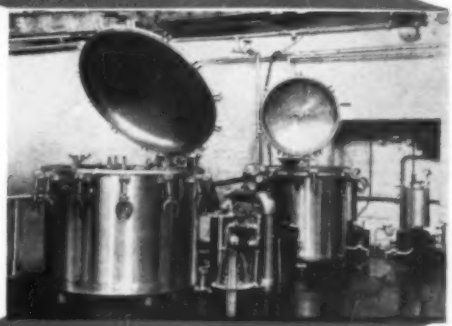

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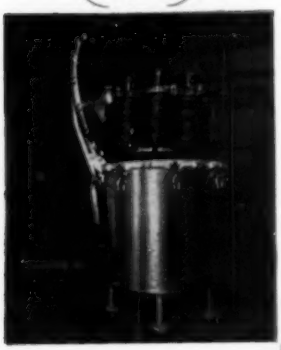

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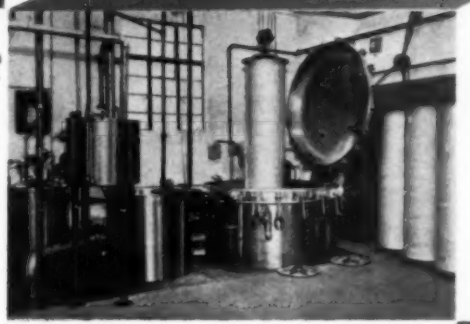

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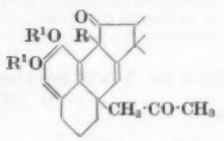
Colour and Constitution. III— Effect of Methyl Substitution on the α- and β-Bands of the Ultraviolet Spectrum of Alternant Hydrocarbons
D. Peters
J.C.S., 4182–4188 (Oct. 1957)

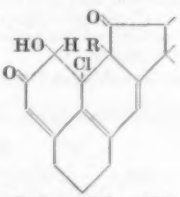
This effect is discussed in terms of the Hückel L.C.A.O. molecular-orbital theory, and it is found that, in order to obtain results as general as those for the *p*-band (cf. *J.C.S.*, 646 and 1993 (1957)), the inclusion of an empirical element is essential. Data are included for naphthalene, anthracene, phenanthrene, chrysene, pyrene, 3:4-benzophenanthrene, and 1:2-benzanthracene, and indicate qualitative agreement between calculated and observed shifts. H.H.H.

Pristimerin. II— Further Reactions involving the Chromophore
P. K. Grant and A. W. Johnson
J.C.S., 4669–4672 (Nov. 1957)

Methylation of the wood pigment pristimerin (cf. *J.C.S.*, 4079 (1957)) by dimethyl sulphate, potassium carbonate, and acetone is shown to involve an addition of acetone to the pristimerin chromophore. This reaction, which is catalysed by acid as well as by alkali, is interpreted as the addition of acetone across the activated methylene–quinone system of pristimerin to give structure I. The red product of addition of HCl to pristimerin contains no ionic chlorine and is formulated as II, in which the main conjugated system of the chromophore is maintained but the strong interaction between the hydroxyl and adjacent carbonyl groups is diminished.



(I: R = O·CH$_3$ or CH$_3$; R^1 = H)



(II: R = O·CH$_3$ or CH$_3$)

H.H.H.

Size and Structure of Commercial and Graphitised Carbon Blacks by Low-angle X-Ray Scattering and Colloid-chemical Methods
K. Inouye
Bull. Chem. Soc. Japan, **30**, 525–531 (July 1957)

Colour Stability of Red Iron Oxide Pigments
Philadelphia Paint and Varnish Production Club, Red Pigments Committee (H. A. McConaghie, Chairman)
Off. Dig. Fed. Paint Varn. Prod. Cl., **29**, 1144–1152 (Nov. 1957)

Eleven iron-based pigments were dispersed by their respective manufacturers and then made into solid colour enamels (pigment volume concn. 15%) in one laboratory. Further series were prepared in which a 15% P.V.C. chalk-resistant TiO_2 enamel was blended with each red enamel to give iron pigment–TiO_2 ratios of 50:50, and 10:90. These 33 enamels were sprayed on to metal panels which had been undercoated with a red oxide primer, and complete sets were exposed in four different locations. The exposed panels were evaluated for chalk-fading and gloss (both polished and unpolished), and the results are reported. It was concluded that all the pigments were of equal colour stability, irrespective of iron content, and none had any adverse effect on film durability. Other factors such as mass- and tint-tone, and ease of dispersion will largely determine which pigment is best suited to a particular product. J.W.D.

Photochemical Changes on Pigment Surfaces
H. F. Clay
J. Oil & Col. Chem. Assocn., **40**, 935–951 (Nov. 1957)

When lead chromes are irradiated with a mercury-vapour lamp CO is evolved; this suggests that oxygen is liberated in the atomic form and not, as suggested by Lashof (*J. Chem. Phys.*, **11**, 196 (1943)) in the mol. form. Carbon-free lead chromes evolve no gas, and the atomic oxygen is probably taken up by Pb ions before it can combine to form mol. O_2, or possibly becomes attached to the glass of the apparatus. The action of the hydrated oxides of Al, Ti, or Si, in improving the light-fastness of chromes appears to be photochemical in character, and to be associated with the distribution of these substances in the form of thin layers. No significant amount of gas is evolved when titania are similarly irradiated, even in the presence of water vapour. Experimental evidence indicates that titania acts photocatalytically in the oxidation of organic matter present; water may play a part in the action. Atomic O does not appear to play any part. It is suggested that the chalking of titania may result from a similar process; there is a review and discussion of available evidence concerning the causes of chalking. Zinc oxide also acts photocatalytically in the oxidation of organic matter, and a gas is evolved during its irradiation which contains 5% CO_2 and 82% O_2. Much less CO_2 is formed than with TiO_2, and it is noted that this accords with the superior chalk-resistance of ZnO. The apparatus used for the collection of evolved gases from irradiated pigments, and for their analysis, is illustrated, and the method is described. The paper contains also useful summaries and discussions of other work on the surface photochemistry of pigments. J.W.D.

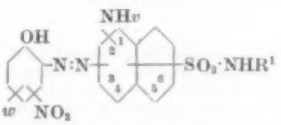
PATENTS

Metal(Chromium and Cobalt)-complex Monoazo Pyrazolone Dyes for Wool, Nylon, etc.
Ciba *BP* 787,305

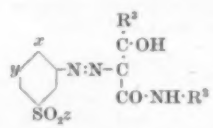
The metal complex containing 1 atom Co to 2 mol. of the monoazo dye 2-aminophenol-5-sulphonamide→3-methyl-1-phenyl-5-pyrazolone is a neutral-dyeing brown-orange dye for wool, nylon, etc., and the corresponding Cr complex is a scarlet. E.S.

Metal(Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.
S *BP* 786,928

Metal-complexes containing 1 atom of Co combined with 2 mol. of monoazo compounds—



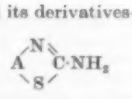
(*v* = H or CH$_3$; *w* = H, Cl, CH$_3$, or NO$_2$; R^1 = 3′-methoxypropyl or 2′-hydroxypropyl; NH*v* is in the 1 or 2 position and is *ortho* to the azo link; SO$_2$NHR^1 is in the 4, 5, or 6 position) or with 1 mol. of such a monoazo compound and 1 mol. of a monoazo compound 2-aminophenolsulphonamide → 2-*m*-chlorophenylaminonaphthalene or—



(*x* = H or OCH$_3$; *y* = H, CH$_3$, or acetylamino; *z* = Alk of 1–4 C, or the radical of a primary or secondary amine; R^2 = Alk of 6–18 C; R^3 = Alk or Ar) dye wool, nylon, etc., blue, green, and olive from neutral or weakly acid baths. Thus 1 mol. of the monoazo compound 2-amino-5-nitrophenol→1-naphthylamine-4-sulphon-β-hydroxypropylamide and 1 mol. of the monoazo compound 2-aminophenol-4-sulphonamide → 2-*m*-chlorophenylaminonaphthalene are heated in formamide at 90–100°C. with $CoSO_4$ to give the complex which dyes wool blue. E.S.

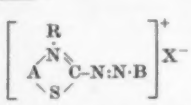
Basic Monoazo Dyes for Polyacrylonitrile Fibres Derived from 2-Aminothiazoles, and 2-Aminobenzthiazoles
Gy *BP* 786,929

2-Aminothiazole and its derivatives—



(A = alkene, e.g. ethene or propene, bound by 2 neighbouring C atoms joined by a double bond, to the S and N atoms; substituents such as phenyl, Hal, NO_2, may be present) are diazotised and coupled with suitable coupling components, especially *para*-coupling arylamines, free of

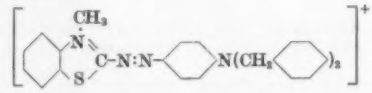
SO_3H and similar groups, and the azo compounds so formed are treated with alkylating agents to give quaternary ammonium salts—



(R = Alk, Aralk, etc.; B = radical of coupling component; X = anion), which dye polyacrylonitrile fibres. Thus 2-aminothiazole is diazotised in nitrosylsulphuric acid and coupled with α-naphthylamine, and the product treated in chlorobenzene with dimethyl sulphate, to give a blue-violet, soluble, basic dye.

BP 787,369

Similar dyes are made by alkylating monoazo dyes of the type 2-aminobenzthiazole→arylamine. Thus 2-aminobenzthiazole is diazotised in nitrosylsulphuric acid and coupled with *NN*-dibenzylaniline. Refluxing in chlorobenzene with dimethyl sulphate then gives the bright blue-violet dye—

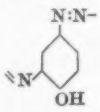


isolated as its zinc chloride double salt. E.S.

Azo Direct Dyes for Aftercoppering

ICI *BP* 786,567

Azo dyes containing the grouping—

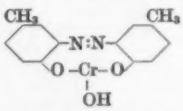


(the N atom *ortho* to the OH group is part of a heterocyclic ring) are converted into *O*-acyl derivatives, using an acylating agent derived from an acid HO·B·X·Z (B = CO or SO_2; X = cyclic residue; Z = COOH, SO_3H, or a quaternary ammonium group) to give copperable direct dyes similar to those of *BP* 761,776 (J.S.D.C., **73**, 66 (1956)), where the acyl group introduced is a sulphato group. Thus the disazo dye 4:4′-diaminobenzanilide → (8-hydroxyquinoline)$_2$ suspended in pyridine is heated with *m*-sulphobenzoyl chloride. The product dyes cellulose orange by an alkaline aftercoppering process. E.S.

Insoluble Metal(Chromium)-complex Azo and Azomethine Dyes for Wool and Nylon

BASF *BP* 787,378

The insoluble complexes containing 1 atom of Cr to 1 mol. of *oo*′-dihydroxy-, *o*-hydroxy-*o*′-carboxy-, or *o*-hydroxy-*o*′-amino-azo- or -azomethine compounds free of SO_3H groups, dye wool and polyamide fibres from fine dispersions at temperatures of 90–130°C. Thus the monoazo compound *p*-cresidine → *p*-cresol is chromed with demethylation of the OCH_3 group to give—

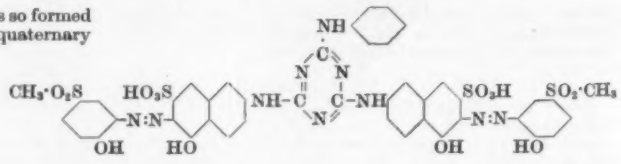


A fine dispersion of the complex applied to wool at the boil in presence of a wax alcohol-ethylene oxide condensate gives a uniform red. E.S.

Red, Copperable Disazo Direct Dyes

S *BP* 786,663

Cyanuric trichloride is condensed with 2 mol. of J acid or *N*-*p*-aminobenzoyl-J acid, the product is coupled with 2 mol. of diazotised *o*-aminophenols, one of which contains an alkylsulphonyl or sulphonamide group, and the third Cl atom may be replaced by condensation with NH_3 or an amine or by treatment with e.g. NaOH to give red direct dyes suitable for aftercoppering. Thus the condensation product of cyanuric chloride with 2 mol. of J acid and 1 mol. of aniline is coupled with 2 mol. of diazotised 2-amino-4-methylsulphonylphenol in aq. pyridine to give—

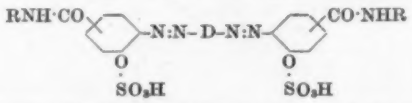


E.S.

Metallisable Disazo Direct Dyes having a Sulphato Group

ICI *BP* 786,745

Sulphuric esters of *o*-hydroxyaminobenzanilides are diazotised and coupled with suitable *ortho*-coupling hydroxy or keto-enol compounds to give disazo dyes—

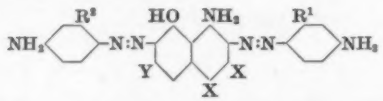


(D has metallisable groups *ortho* to the azo links; and is free of COOH or SO_3H groups; R = subst. or unsubst. phenyl) which give metal complexes on the fibre with displacement of the sulphato groups. Thus 3-nitro-4-hydroxybenzanilide is added to a cold mixture of pyridine and chlorosulphonic acid in ethylene dichloride, and warmed to 50–55°C., to give the sulphuric ester. Reduction with Fe then gives 3-amino-4-hydroxybenzanilide, which is diazotised and coupled with 4:4′-bis(3″-methylpyrazol-5″-one-1″-yl)diphenyl to give a disazo compound which dyes cotton reddish yellow, converted to yellow brown on aftercoppering. E.S.

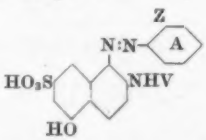
Grey, Hexakisazo Direct Dyes for Aftercoppering

S *BP* 786,670

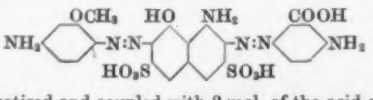
Diaminodisazo compounds—



(R^1 and R^2 = OCH_3 or COOH; one X = H, the other X = SO_3H; Y = H or SO_3H) are tetrazotised and coupled with 2 mol. of aminoazo compounds—



(Z = OH or COOH; V = Alk, Ar etc.; A may contain common substituents) to give hexakisazo dyes which give greys on cellulose by an aftercoppering process. Alternative syntheses are described. Thus the diaminodisazo compound—



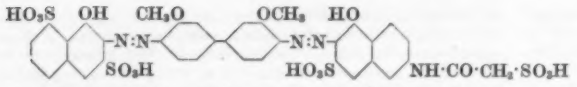
is tetrazotised and coupled with 2 mol. of the acid-coupled monoazo compound 2-aminophenol-4-sulphonamide → *N*-phenyl-J acid in aqueous ammoniacal pyridine.

E.S.

Blue and Grey Metal(Copper)-complex Disazo Direct Dyes

Gy *BP* 786,918

The copper complexes of disazo compounds made by coupling a tetrazotised benzidine, containing substituents in the 3:3′ positions capable of complex formation, with 2 mol. of *ortho*-coupling naphthols at least one of which contains the group –NH·CO·CH_2·SO_3H attached preferably directly to the naphthalene nucleus, are blue or grey direct dyes. Thus *o*-dianisidine is tetrazotised and coupled in presence of Na_2CO_3 with 1 mol. of 1-naphthol-3:8-disulphonic acid. A soln. in aq. Na_2CO_3 of *N*-*w*-sulphoacetyl-J acid is then added, to give the disazo compound—

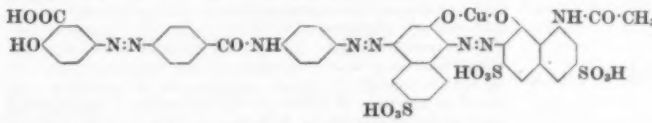


Heating at 90–95°c. with ammoniacal $CuSO_4$ gives the complex which dyes cellulose blue. E.S.

Green and Olive Metal(Copper)-complex Trisazo Direct Dyes

FBy *BP* 787,416

Soluble copper-complex dyes are made by coppering a trisazo compound B ← A → C → D (A = residue of tetrazo compound of diphenyl or benzanilide series; B = residue of an *o*-hydroxybenzoic acid; C = residue of an alkoxynaphthylaminemonosulphonic acid; D = residue of a 1:8-aminonaphtholdisulphonic acid) to give a substantially insoluble complex containing 2 atoms Cu per mol., which is heated with a complex-forming alkylamine or alkylaminecarboxylic acid, whereby the Cu atom attached to the *o*-hydroxybenzoic acid residue is removed. The products are green and olive-green direct dyes which may be aftercoppered. Thus 4:4′-diaminobenzanilide is tetrazotised and coupled first with 1 mol. of salicylic acid and then with 1 mol. of 2-ethoxy-1-naphthylamine-6-sulphonic acid, and the aminodisazo compound so formed is diazotised and coupled with 1 mol. of *N*-acetyl-H acid. The trisazo compound so formed is heated at 110–125°c. with aq. $CuSO_4$ giving the insoluble copper-complex converted into the soluble complex—



by treatment at 80°c. with aq. ethylene-bis(iminodiacetic acid). It dyes cellulose olive which may be aftercoppered. E.S.

Thiaxanthone Quinone

FBy *BP* 786,925

Early attempts to oxidise 1:4-dihydroxythiaxanthone to 1:4-thiaxanthonequinone gave only undefined polyhydroxy compounds (*J.C.S.*, **99**, 1538). The present method gives the 1:4-thiaxanthonequinone as brown, flat needles m.p. 237°c. K bromate (2 g.) dissolved in water (30 g.) is added at room temperature with stirring to 1:4-dihydroxythiaxanthone (3·2 g.) and glacial acetic acid (80 ml.). After 3 min. stirring at room temperature and cooling with iced water the precipitate (1·6 g.) is filtered, water washed and dried. E.T.

Copper Complexes of α-Hydroxyanthraquinones; Disperse Dyes

ICI *BP* 787,284

Green, blue, violet or brown copper complexes of α-hydroxyanthraquinones having at least one $-NH_2$, –NH Alk or –NH Aryl group in an α-position not occupied by an –OH group are pigments or disperse dyes for synthetic fibres. Suitable α-hydroxyanthraquinones are 1-anilino-4-hydroxy-, 3-bromo-1-anilino-4-hydroxy-, 4-nitro-5-anilino-1:8-dihydroxy-, and 3:4-pyridino-2-hydroxy-anthraquinones. These are treated in a solvent, preferably the water-miscible 2-ethoxyethanol, with a solution or dispersion of a Cu salt, e.g. Cu acetate, or a Cu complex, e.g. Cu acetylacetone. Reaction takes place in 1–2 hr. at the boil and the insoluble products are obtained by filtration. Dyeings on synthetic fibres have good light and wash fastness and superior sublimation fastness when compared with that of the parent anthraquinone compounds. E.T.

Halogenated Diamino-Dihydroxy Anthraquinones—Blue Disperse Dyes

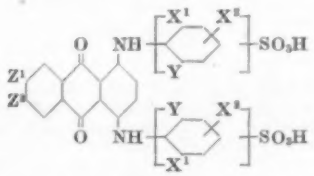
FBy *BP* 787,379

Blue disperse dyes of good light fastness and suitable for dyeing polyester fibres are produced by halogenating 4:8-diamino-1:5-dihydroxy or 4:5-diamino-1:8-dihydroxy-anthraquinones. Dyes containing 5–40% Br or 3–25% Cl are preferred. They are made by the action of the halogen itself or a halogenating agent, e.g. SO_2Cl_2, on the diamino-dihydroxyanthraquinone in dilute to fuming H_2SO_4 or conc. HCl or an organic solvent, e.g. nitrobenzene or trichlorobenzene. Boric acid and halogenation catalysts, e.g. iodine, may be added and reaction takes place at about 50°c. Up to 4 Hal atoms per anthraquinone mol. can be thus introduced. E.T.

Anthraquinone Acid Dyes

Gy *BP* 787,299

Reddish-blue dyes giving dyeings of good wet and light fastness on wool dyed from a neutral or slightly acid bath and also suitable for other natural or synthetic polypeptide fibres are of formula—

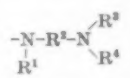


(X^1 or X^2 = phenyloxy, phenylthio or naphthyloxy whilst the other X = H or Alk; Y = Alk; Z^1, Z^2 = H or Hal). They are prepared from anthraquinone derivatives which have replaceable 1:4-substituents, e.g. 1:4-dichloro-, 1:4-dibromo and 1:4-dihydroxy-anthraquinones, and which may also have 6- and/or 7-substituents such as F, Cl, Br, arylthio and alkylsulphonyl groups. These compounds are condensed with 6-alkyl-1-amino-benzene aryl ethers or thioethers which have in the 2-position of the amino-substituted benzene ring an Alk, ether or thioether group. As condensation media the lower fatty acids or higher boiling alcohols, e.g. butanol, or ethyleneglycolmonomethylether may be used along with acid binding agents and catalysts, e.g. Cu, or partial reduction of the quinone groups may be employed. The condensation product is then sulphonated with conc. H_2SO_4, oleum, $ClSO_3H$ etc. E.T.

Basic Phthalocyanine Dyes

FBy *BP* 784,843

Basic dyes for cotton, wool, polyamide or polyacrylonitrile fibres are phthalocyanine derivatives containing 1–4 radicals of formula—

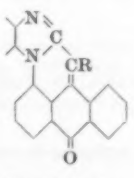


(R^1 = H or Alk; R^2 = acyclic bivalent radical; R^3, R^4 = aliphatic radicals or may form a ring with the N atom). A phthalocyanine dye with 1–4 replaceable halogen atoms is reacted with a tertiary amine $HR^1NR^2NR^3R^4$ *or* it is reacted first with an aliphatic primary or secondary amine containing at least one Hal atom and then further reacted with a secondary amine. The replaceable halogen in the phthalocyanine compound may be present in such groups as –COCl, $-SO_2Cl$ or ω-chloromethyl. Suitable Hal containing amines are β-chloroethylamine, β,γ-dibromopropylamine and suitable secondary amines are dimethylamine, piperidine and pyrroline. Condensation is effected in excess of the base or in an inert organic solvent or in aqueous suspension at 0–100°c. in presence or absence of an acid-binding agent. E.T.

Anthraquinone Vat Dyes

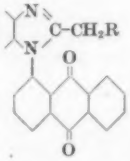
Ciba *BP* 787,311

Yellow vat dyes suitable for dyeing or printing in the usual manner or in the form of their leuco-ester salts contain the grouping—

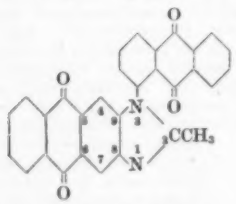


(R = a substituent; the 2 N atoms are advantageously attached to a second anthraquinone nucleus; the anthraquinone nuclei may contain further substituents). They

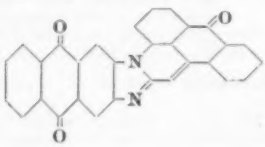
are produced as follows—(*a*) a 1-(*o*-amino-arylamino)-anthraquinone is condensed with R–CH_2–COOH or a functional derivative e.g. chloroacetylchloride; (*b*) the product is ring-closed with e.g. conc. H_2SO_4 to give—



(*c*) this is further ring-closed under alkaline conditions. Stages (*a*) and (*b*) can be carried out without an intermediate separation. Thus, 2-amino-3:1′-dianthrimide (7), Ac_2O (60) are heated to 100°c. and conc. H_2SO_4 (10) added. Stirring at 105–110°c. until dissolved, precipitation into water and neutralisation with NH_4OH give 5:6-phthaloyl-2-methyl-3-(1′)-anthraquinonyl-benzimidazole—



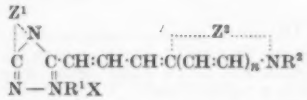
This compound (5), ethyl alcohol (150), 35% NaOH (3) boiled for 1 hr. gave the yellow vat dye—



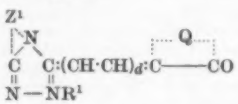
E.T.

Cyanine Dyes
Kodak *BP* 783,021

Dyes of formula—



(R^1 and R^2 = subst. or unsubst. Alk or aralkyl; X = acid radical; n = 0 or 1; Z^1 = atoms to complete a benzothiazole or quinoline nucleus; Z^2 = atoms to complete a 5- or 6-membered ring) or



(d = 1 or 2; Q = atoms to complete a 5- or 6-membered heterocyclic ring) e.g. 2′:3-diethyloxa-1′-*s*-triazolo[4,3-*a*]-quinocarbocyanine iodide, are used as photographic sensitisers. C.O.C.

Water-dispersible Titanium Dioxide (C.I. Pigment White 6)
National Lead Co. *USP* 2,744,029

Calcined titanium dioxide is wet milled, the milled slurry flocculated in presence of a volatile organic acid and then heated to drive off the acid. The resulting pigment has an unusually high degree of dispersion in water. C.O.C.

Titanium Dioxide—Alkaline Earth Metal Sulphate Pigments
National Lead Co. *USP* 2,744,028

An alkaline earth metal sulphate slurry is boiled with 5–50% of the aqueous solution of titanium sulphate necessary to produce the desired composite pigment. The remaining portion of the titanium sulphate solution is then added and the whole further boiled. The precipitate is filtered off, washed and calcined at 900–1000°c. to yield a composite pigment in which the TiO_2 is in the rutile form. C.O.C.

Ultramarine (C.I. Pigment Blue 29)
IC *USP* 2,745,766

Sodium aluminate, silica, sulphur, a reducing agent (e.g. pitch or an alkali metal organic compound), and a sodium salt (e.g. Na_2CO_3 or Na_2S) are heated under non-oxidising conditions at > 600°c. for 20–180 min. The mass is then cooled to 450–800°c., slowly oxidised with air and/or SO_2 or NO_2 for 30–180 min., washed and ground. This yields a superior product of consistent quality. C.O.C.

Polymerisation of Dyes in Solution (VIII p. 104)
Colour Photography (IX p. 107)

V—PAINTS; ENAMELS; INKS

Dispersion, Distribution, and Binding of Pigments in Emulsion Paint Films
W. C. Prentiss
Off. Dig. Fed. Paint Varn. Prod. Cl., **29**, 951–965 (Oct. 1957)

It is demonstrated that an ideal system of spherical pigment particles completely surrounded by spherical latex particles (the critical pigment volume concentration), 25% of the particles will be of pigment. Practical conditions are non-ideal—(i) pigment particles are of irregular shape and vary widely in size; (ii) the dispersion may not be stable enough to permit mobility of all particles until the final moment of packing; and (iii) particle aggregation may occur as the concn. increases during drying. There is discussion of these factors and of the steps that may be taken to minimise them. Evidence of the importance of pigment dispersion and distribution upon paint properties is reviewed; there is loss of gloss at pigment volume concentration > 8% with emulsion paint films containing only TiO_2, whereas the theoretical critical pigment volume concentration is 25%. A dispersant should (i) enable the pigment aggregates to be wetted by the continuous phase; (ii) assist the deflocculation of these aggregates; and (iii) stabilise the dispersed pigment particles against reflocculation or aggregation. No single agent will fulfil all these functions. Some of the dispersants commercially available are referred to, and their effects described. A three-component dispersant may be used, e.g. a nonionic wetting agent such as a polypropylene oxide, a synthetic anionic deflocculant such as a polyphosphate, and a polymeric stabiliser such as a low-mol.wt. polyacrylic acid salt. A triangular diagram may be used to determine the composition of a three-component agent designed to yield a required level of some such property as gloss. There are five methods for selecting the amount of dispersant required (including trial and error); these are briefly reviewed. J.W.D.

Dispersion of Carbon Black (C.I. Pigment Black 6 and 7)
L. J. Venuto
J. Oil & Col. Chem. Assocn., **40**, 725–736 (Sept. 1957)

Particles of carbon black usually have diameters within the range 10–83 μ. and they exist in aggregates of more than 4×10^{12} units. Dispersion consists in the tearing apart of these aggregates to form microflocculates that do not re-aggregate; grinding does not reduce particle diameter. The pretreatment of the carbon black by the user, the process of milling and the assistants used, and the incorporation of the dispersed base-paste into a high-quality car enamel of good stability, are discussed in detail. J.W.D.

Pigment Dispersion. VI—Phthalocyanine Blue
New York Paint and Varnish Production Club Technical Subcommittee No. 53 (J. J. Oates, Chairman)
Off. Dig. Fed. Paint Varn. Prod. Cl., **29**, 1113–1143 (Nov. 1957)

The investigation is discussed under four headings—(i) nature of the Cu phthalocyanine blue pigment (I); (ii) practical dispersion procedures; (iii) evaluation of dispersion

efficiency, and maintenance of this efficiency on admixture with a TiO_2 base; and (iv) behaviour of I–TiO_2 in terms of the pigment–resin–volatiles relationship. The following conclusions are reached— (i) there is a practical min. amount of dispersion work which provides the optimal economic return when I is used for tinctorial purposes; (ii) this practical work min. is best measured by tint-strength evaluation and not by the gauge measurement of fineness of grind; (iii) tint-strength measurement is reliable only if there is a wide range of compatibility in the pigment–binder–volatiles system; (iv) a good mechanical procedure for blending I with a white is essential if the full strength of the dispersion is to be realised and retained; (v) a short run in a steel ball-mill with high loading is the most economical method of dispersion, and gives the greatest ease in handling; (vi) pigmented systems may be regarded as complex when they contain more than one pigment or liquid component. Even "simple" vehicles such as linseed oil or alkyd resins are in themselves complex. It is suggested that practical knowledge of the consistency characteristics of both the colour base paste and the white tinting base should be developed since this assists in establishing the best incorporation procedure. J.W.D.

Revaluation of the Function of Pigments in Paints
A. C. Elm

Off. Dig. Fed. Paint Varn. Prod. Cl., **29**, 351–385 (April 1957)

A review, under the following headings— hiding power and colour; yellowing of interior finishes; water resistance and washability; seeding; chalking of exterior paints; metal protective paints. Recent evidence confirms established theories in these connections. There are 14 graphs, diagrams, and photographs. J.W.D.

Hiding Power of Titanium Pigments
F. B. Stieg

Off. Dig. Fed. Paint Varn. Prod. Cl., **29**, 439–452 (May 1957)

Recommendations are given for the securing of max. hiding power, with economy, using specific American grades of TiO_2 (C.I. Pigment White 6). For max. economy at any desired hiding level, pigmentations should be adjusted to the highest practicable pigment volume concentration by the use of a calcium-base TiO_2. Minimal wet-to-dry loss of hiding power or tinting strength is obtainable both at the lowest possible pigment volume concentration and at the highest possible solids content. J.W.D.

Selective Polar Adsorption
L. Dintenfass

Chem. and Ind., 560 (4 May 1957)

The adsorption of polar organic molecules from organic solvents such as toluene, benzene, acetone, white spirit, etc. on to the surface of rutile titanium dioxide, talc, $BaSO_4$, and other pigments and extenders has been studied. Selective polar adsorption, which is distinctly different from purely physical adsorption of non-polar substances, and from chemisorption, can take place only when the active area of the adsorbent corresponds to the polar group of the adsorbate. Adsorption patterns and governing principles emerging from the study are detailed under 12 headings. J.W.D.

Rheological Properties of Stable Suspensions of Very Small Spheres at Low Rates of Shear
G. F. Eveson

J. Oil & Col. Chem. Assocn., **40**, 456–477 (June 1957)

The relative viscosity (R.V.) of Newtonian stable suspensions is independent of mean particle size for spheres larger than ca. 15 μ.; with smaller spheres relative viscosity increases with decreasing mean particle size. This effect cannot be explained in terms of solvation shells of dispersion medium surrounding the particles. Non-Newtonian behaviour is associated with incomplete dispersion of the particles and can be eliminated by adding sufficient dispersing agent to produce complete dispersion. Microscopic examination of non-Newtonian systems reveals lattice structures developed within a suspension at rest, tending to prevent the relative motion of single particles or groups of particles. These structures are broken down by shearing but they reform when the system is left to stand. A rotational viscometer used at low rates of shear ($< \sim 1{\cdot}0$ sec.$^{-1}$) will detect sensitively the onset of flocculation in a suspension. For a given ζ-potential flocculation occurs when the distance between spheres is less than a certain critical value; this critical distance decreases with increase in ζ-potential. Measurement of the electrical conductivity of a suspension may afford a means of defining the degree of dispersion of the particles. J.W.D.

Finishes for Heating Appliances— Epoxide Paint and Vitreous Enamel compared
N. S. C. Millar

J. Oil & Col. Chem. Assocn., **40**, 478–487 (June 1957)

Epoxide paints and vitreous enamels are compared with reference to finish, initial brightness, colour and gloss, colour and gloss stability on ageing, adhesion, surface hardness, heat-resistance and chemical resistance. Vitreous enamel has the sole disadvantage of poor chip-resistance, whereas epoxide–urea–formaldehyde resin systems have very poor resistance to abrasion and heat. J.W.D.

Flotation
R. J. Cole

J. Oil & Col. Chem. Assocn., **40**, 741–744 (Sept. 1957)

Stokes's law applies to systems of lower concn. than paints, but it remains, for these, sufficiently valid to indicate a tendency. The mobility of pigment particles in a binder soln. is given, in these terms, by the function $d^2(\varrho - \varrho_0)$ or, for oil media, $d^2(\varrho - 0{\cdot}95)$, where d = particle diameter, ϱ = particle density, and ϱ_0 = the density of the medium. Values range from 10^{-5} for a fine colour carbon black, to 18·0 for certain white leads. The greater the difference between the values associated with different pigments, the greater the liability to flotation in a mixed pigment system. Uneven flotation leads to the formation of patterns (frequently regular hexagons); these are produced by a circulating cell system caused by turbulence. The main factors are those of different pigment particle mobilities, and temperature gradients (with associated density differences) in the laid wet film. Silicone oil suppresses such effects in a manner similar to that of the action of oil on a stormy sea. J.W.D.

Wet and Dry Opacity of Paints
D. M. James

J. Oil & Col. Chem. Assocn., **40**, 786–789 (Sept. 1957)

Paints lose opacity on drying because as solvent is lost the refractive index of the medium rises; the effect of increased pigment concn. appears to be small. For a given pigment the value of the critical pigment vol. concn. above which the paint gains opacity on drying, depends on the nature of the medium.

F. B. Stieg

Ibid., 794–796 (Sept. 1957)

The author defends certain of his views (*Off. Dig. Fed. Paint Varn. Prod. Cl.*, 701 (1956), 439 (1957)) against criticisms embodied in the paper by D. M. James (abstract above). In addition to criticisms of the experimental methods used by James, the author maintains that it is incorrect to assume that loss of gloss may be taken as an indication that the critical pigment vol. concn. has been reached; it may be caused, even at low pigment vol. concn., by flocculation of pigment. It may also be due to the presence of large pigment particles in a thin film.

D. M. James

Ibid., 884–885 (Oct. 1957)

The author replies to the criticisms of F. B. Stieg (above abstract). J.W.D.

PATENTS

Storage of Aluminium Pastes
Aluminium Co. of America *BP* 772,507

The development of gas pressure caused by presence of moisture in sealed containers of pastes, paints, inks etc. containing C.I. Pigment Metal 1 is reduced by adding 0·25–51% of a nitroparaffin, nitrotoluene or nitroxylene. E.C.

Complex Aluminium Alkoxides
Hardman & Holden *BP* 772,144

Aluminium alkoxides, useful as thickening agents for alkyd resins and as cross-linking agents for polyester and polyamide resins, are prepared by heating an aluminium alkoxide derived from an alcohol of 3–10 C (2 mol.) with a glycol (1 mole: 5 C atoms) or a polypropylene or polybutylene glycol (6–40 C) in presence of a solvent, e.g. white spirit, to recover alcohol from the alkoxide. E.C.

Pigmented Rolled Masses for Use in Lacquers
Deutsche Gold- und Silber-Scheideanstalt *BP* 784,512

An aqueous dispersion of a pigment or filler is intensively stirred with an aqueous dispersion of a film-former (i.e. cellulose derivative or synthetic resin) to cause the pigment or filler to deposit on the particles of the film-former. A gelatinising agent is then added to the dispersion, most of the water separated off and the product worked up into a rolled mass. On cooling the pigmented mass is easily chipped, the chips being readily soluble in lacquer solvents.
C.O.C.

Magnesite Cement Coating Compositions
Deutsche Magnesite *BP* 772,017

Magnesite cement coatings for wood, fabric and building materials resist peeling and cracking if 2–3% of a water-soluble urea-, melamine-, or phenol-formaldehyde resin or of unspecified vinyl polymers is added either to the magnesia or the magnesium chloride component before mixing.
E.C.

VI—FIBRES; YARNS; FABRICS

Universal Decimal Classification of Fibres and Textiles
J. Brämer
Faserforsch. und Textiltech., **8**, 368–372 (Sept. 1957)

Surface of Cotton Fibres
V. W. Tripp, A. T. Moore, and M. L. Rollins

I— Native Fibres
Text. Research J., **27**, 419–426 (June 1957)

The topography of unmodified cotton fibres has been examined microscopically by means of surface replica techniques. The outstanding characteristic of the surface is a system of roughly parallel ridges and grooves spiralling around the fibre at an acute angle to its axis. The pattern of the surface is a reflection of the spiral fibrillar structure of the cellulose beneath the primary wall of the fibre. The surfaces of most native cottons are quite similar, although statistically significant differences exist between certain samples. The surfaces of fibres from unopened bolls are relatively smooth, but assume the roughness of the typical fibre on drying for the first time. A characteristic surface formation appears to be associated with fibres known to have undergone compression.

II— Modified Fibres
Ibid., 427–436

Microscopical studies of surface replicas of chemically finished or modified cotton fibres have shown that the characteristic surface of native cotton is often altered. While removal of the wax from the fibre surface has little effect, scouring usually uncovers the fine cellulose fibrils of the primary or even secondary walls. Mercerisation does not eliminate the surface roughness of the cotton fibre. Additive finishes, including starch, carboxymethyl cellulose, colloidal silica and acrylic polymer resins change the fibre topography to an extent dependent on the amount applied, in general filling the grooves when applied at low concentrations but covering most of the surface at higher concentrations. Chemical modifications which increase the fibre cross-sectional area make the surface smoother, but extensive swelling during preparation appears to create roughness in some modifications. Crease-resistant finishes do not alter the fibre topography significantly. S.B.D.

Degradation of Cotton by Heat
M. Aubry
Bull. Inst. Text. France (66), 53–66 (April 1957)

Samples of cotton were treated in air at temperatures in the range 90–150°C. for times ranging from 1 to 96 hr. The following properties of the treated and untreated samples were investigated— (*a*) microscopical appearance (*b*) moisture relations, (*c*) swelling, (*d*) resistance to extension, (*e*) D.P., (*f*) alkali solubility, (*g*) pH of aqueous extract, (*h*) copper number, (*i*) response to Muller's reaction (*ibid.*, (3), 139 (1947)), and (*j*) dyeing affinity. Results obtained are compared with previous literature reports. The nature of the chemical changes produced by heat are discussed. 37 references to the literature are given.
J.C.F.

Comparative Study of the Behaviour at High Temperatures of Partially Acetylated Cotton and some other Fibres
R. Specklin
Bull. Inst. Text. France, (66), 67–94 (April 1957)

Investigation of the stability to heat of various natural, modified natural, and man-made fibres confirms the superior properties of partially acetylated cotton. Partially acetylated wool shows no improvement over the untreated fibre. Polyamide fibres are rapidly damaged by heat, but polyacrylate and polyester fibres are more resistant. Use of the concept of "time of half-destruction" is suggested as a basis for comparing the stabilities of different fibres.
J.C.F.

Crystallite Orientation in Jute treated with Caustic Soda Solution
V. D. Gupta
J. Polymer Sci., **26**, 111–112 (Oct. 1957)

X-Ray studies show that there is a much better degree of restoration in crystallite orientation when jute treated with caustic soda solution is washed with boiling water instead of water at room temperature. There is an improvement in orientation when the fibre is allowed to dry under tension after being washed, particularly if the fibre has been washed with boiling water before drying.
W.R.M.

Viscose Ripening
H. L. Vosters
J. Polymer Sci., **26**, 242–244 (Nov. 1957)

Careful measurements of the ripening of different viscoses suggests that in viscose solution there are two types of xanthates, one ripening at a first-order and the other at a second-order rate. W.R.M.

Recovery of Wool from Sheepskin Pieces
J. Millet and J. Barut
Bull. Inst. Text. France, (65), 41–50 (Feb. 1957)

On the basis of laboratory work, the following procedure has been found satisfactory— (i) sweating for 24 hr.; (ii) immersion in water at 70–80°c. for 1 hr.; (iii) treatment with enzyme solution (8–10 g. of Rapidase per litre) at 40°c. for 36 hr., liquor ratio 10–15:1, pH 6–6·5; (iv) washing in soap–soda. J.C.F.

Models of α-Keratin Structure
R. D. B. Fraser, T. P. MacRae, and D. H. Simmonds
Biochim. Biophys. Acta, **25**, 654–655 (Sept. 1957)

The amino-acid composition and density of descaled porcupine quill tip have been determined. An attempt is made to apply the results of X-ray diffraction studies with the results obtained. P.G.M.

Influence of Ionising Radiations on Wool Fibre Properties
R. A. O'Connell and M. K. Walden
Text. Research J., **27**, 516–518 (July 1957)

Dyed and undyed samples of wool were exposed to soft X-rays, $_{60}$Co γ-rays, cathode rays, and ultraviolet radiation. The effects were evaluated by stress-strain measurements and supercontraction tests. Although slight at low levels of dosage, the damage to the wool fibres was found to be progressive with increases in the radiation. No evidence of the formation of new cross-linkages was discovered. The dyed fibres were found to be more resistant than the undyed fibres to irradiation. S.B.D.

Preparation of Soluble Modification of Keratin
A. Berlin, E. Kirina, and L. Kuznetsova
Myasnaya Ind. S.S.S.R., **27** (6), 48–51 (1956): *Chem. Abs.*, **51**, 8463*h*:
J. Amer. Leather Chem. Assocn., **52**, 640 (Nov. 1957)

Amino-acid Sequence in a Fraction of Fibroin
F. Lucas, J. T. B. Shaw, and S. G. Smith
Biochem. J., **66**, 468–479 (July 1957)

A study has been made of the reaction product of soluble silk fibroin and chymotrypsin. The sequence of the amino-acid residues has been investigated by partial hydrolysis, followed by separation of the products by chromatography and quantitative estimation of their amounts. P.G.M.

Application of the Oil-immersion Method to the Analysis of a Striation Fault in a Wool–Polyester Fibre Fabric
R. Schneider
Bull. Inst. Text. France (65), 53–57 (Feb. 1957)

Striations in a 45:55 wool–delustred polyester fibre fabric were traced to irregular distribution of the components

accentuated by differences in yarn count. The results of the microscopical examination were substantiated by chemical analysis and determination of yarn counts.
J.C.F.

Bulked Yarns Name Index
Anon.
Silk and Rayon, **31**, 1192, 1194–1196 (Nov. 1957)

Profiled Fibres of Special Cross-section and Surface
H. Böhringer
Z. ges. Textilind., **59**, 691–696 (5 Sept. 1957); *Silk and Rayon*, **31**, 728–731 (July 1957); *Modern Textiles*, **38**, 70, 86 (Nov. 1957)
Some defects of conventional melt-spun fibres of circular cross-section and smooth surface are listed. Methods for modifying the shape of the cross-section and the character of the surface are outlined. W.R.M.

Anomalously Low Densities of some Oriented Samples of Poly-6-caproamide
F. Rybnikar
J. Polymer Sci., **26**, 105–106 (Oct. 1957)
Anomalously low densities of drawn samples of poly-6-caproamide are ascribed to submicroscopic holes which arise during drawing. W.R.M.

Polyamide and Polyester Fibres— Recent Progress
G. M. Richardson, H. E. Stanley, and W. W. Heckert
Text. Research J., **27**, 529–539 (July 1957)
Many of the newer processes designed to give new fibre fabrics more useful properties are briefly described. The processes include increasing the bulk and covering power of both filament and staple yarns and controlling their feel and handle; giving fabrics different covering powers, dimensional stability and permanent creases; and reducing pilling both for fabrics and rugs. A section on automatic wash-wear, where tumble-drying replaces ironing, is included. S.B.D.

PATENTS

Adding Sulphur Dyes to Viscose Spinning Solutions
CFM *BP* 782,920
Viscose is mixed with an aqueous solution of a sulphur dye, e.g. C.I. Sulphur Black 1 treated with sodium sulphite. The xanthogenate reduces the dye so that a homogeneous solution is quickly formed. This solution is then extruded in the usual way and during the processing of the filaments the dye is re-oxidised. This yields very evenly dyed filaments of strength the same as those of corresponding undyed filaments. C.O.C.

Carbon-filled Polyamides
DuP *BP* 784,254
Polyamides formed from monomers to which an aqueous dispersion of carbon has been added while they were being polymerised at the boil are much more resistant to weathering than unfilled polyamides. They can be processed in the normal way, e.g. extruded as filaments or films, and can be used for any purpose for which black filaments, etc. are required. C.O.C.

Alkaline Degradation of Polysaccharides (VII this page).
Swelling and Solution of Synthetic Fibre-forming Polar Polymers in Liquids (XIII p. 110)
Measurement of Molecular Orientation in Filaments of Polyethylene Terephthalate using Dichroic Dyes (XIV p. 116)
Measurement of Hydroxyl and Carboxyl End-groups in Polyethylene Terephthalate (XIV p. 116)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Micelles. III— Comparison of Spectral and Other Results
H. Sasaki
Bull. Chem. Soc. Japan, **30**, 326–331 (June 1957)
As a result of spectral observations the author had concluded (*ibid.*, **30**, 186 (1957)) that in aq. soln. of surfactants, for a wide range of surfactant concn. above the critical micellar concn., the concn. of micelles remains constant, only the association number of the micelles increasing. This conclusion is now shown to agree with the results of other authors for measurements of dye solubilisation, conductivity, diffusion, X-ray diffraction, density, viscosity, and osmotic or cryoscopic behaviour. A.J.

Alkaline Degradation of Polysaccharides
I— Soluble Products of the Action of Sodium Hydroxide on Cellulose
G. N. Richards and H. H. Sephton
J.C.S., 4492–4499 (Nov. 1957)
The major soluble products of the degradation of cotton hydrocellulose by boiling oxygen-free 0·5N-NaOH are determined, and the following acids identified— β-D-gluco*meta*saccharinic, αβ-D-gluco*iso*saccharinic, βγ-dihydroxybutyric, C_3 *meta*-saccharinic, αβ-dihydroxy-α-methylpropionic, glycollic, α-D-glucosaccharinic, lactic, dihydroxybutyric, lactyl-lactic, formic, and acetic. D-Gluco*iso*saccharinic and formic acids are produced in greatest amount under the conditions used, which corresponded approximately to those of rayon pulp refining. Cotton hydrocellulose was chosen as a substrate, because its method of preparation and high crystallinity give maximum probability of normal end-groups to each mol., and chemical uniformity along the chains. The rapid formation of γ-lactones in presence of a sulphonic acid-substituted resin, and subsequent partial separation of lactonisable from non-lactonisable acids, are noteworthy.

II— Alkali-stable Residue from the Action of Sodium Hydroxide on Cellulose
G. Machell and G. N. Richards
Ibid., 4500–4506
The alkali-stable polysaccharide resulting from the treatment of cotton hydrocellulose with hot dil., oxygen-free NaOH has been partially hydrolysed in N-HCl at 100°C., and D-gluco*meta*saccharinic acids have been isolated and identified, while α-D-glucosaccharinic acid has been tentatively identified by paper chromatography. It is concluded that these acids originate by alkaline rearrangement of the reducing end-group of the cellulose mol., and so impart stability to alkali. In view of the extensive losses of α-cellulose which occur in the hot alkali refining of wood-pulps, there is technological interest in the nature of the alkali-stable material. H.H.H.

Neutralisation of Acid-treated Wool by Solutions of Sodium Carbonate
R. P. Harker
J.S.D.C., **73**, 554–560 (1957)
Neutralisation of acid-treated wool by solutions of sodium carbonate has been examined by measurement of hydrogen-ion concentrations. The effects of concentration, time, liquor ratio, and temperature on the equilibration process have been studied. A qualitative interpretation of results is given, and a quantitative treatment employing the Donnan theory of membrane equilibria has been examined. AUTHOR

Washing of Man-made Textiles
O. Viertel
Melliand Textilber., **38**, 1023–1029 (Sept. 1957)
The important factors in washing— dirt, textile material, washing agent, and conditions of washing— are discussed. Results of wearing and washing trials have shown that, contrary to popular belief, it is easier to remove dirt and natural soiling matter from synthetic than from natural fibres such as cotton. The importance of temperature and pH of the wash liquor, and the effectiveness of various cleaning agents such as soap and synthetic detergents, are discussed. The text is illustrated with electron micrographs. L.A.T.

Influence of Dechlorination on the Degradation of Fabrics by Repeated Bleaching
A. Fresco and A. Parisot
Bull. Inst. Text. France (66), 7–14 (April 1957)
Cotton and linen fabrics, after hypochlorite bleaching, were given the following alternative treatments— (1) immediate drying, (2) drying after exposure to light and air for 2 hr., (3) drying after freeing from excess hypochlorite by thorough rinsing, (4) drying after dechlorination with metabisulphite. The bleaching and drying cycles were repeated up to 60 times, and samples were taken at intervals for measurement of degradation from (*a*) D.P. and (*b*) wet and dry tearing strengths. In all cases degradation increased as the No. of treatments increased, but the extent of degradation was much greater for treatment (2), whilst the differences between the other three treatments were not significant. Thus if fabric cannot be dried immediately after bleaching, dechlorination by some

means is desirable. Treatment with metabisulphite produces no additional degradation. J.C.F.

Practical Causes of the Chemical Degradation of Rayon and Properties of Modified Celluloses
J. Pinte

Bull. Inst. Text. France (66), 15–40 (April 1957)

Acid treatments lead to the formation of hydrocelluloses, whereas oxycelluloses are formed during oxidising treatments, and by the use of sizes containing drying oils. Desizing with non-ionic detergents in neutral solution produces less strength loss than the use of soap. The effect of traces of metals on the degradation produced by various bleaching treatments is described and discussed, and it is shown that sodium chlorite bleaching is particularly insensitive to the presence of metals. In the second part of the paper the author discusses the properties of modified celluloses with particular reference to alteration in affinity for dyes. It is shown that chemical modification of cellulose produces little change in the macromolecular structure. J.C.F.

PATENTS

Stain Removal
H-E. R. Schmidt *BP* 784,347

Stains caused by non-drying oils or by aqueous or albuminous liquids are removed by covering them with dry powdered magnesium polysilicate and leaving them until the powder absorbs the stain. The polysilicate must contain < 0·5% by wt. of carbonate and 90% of it must pass through a screen of 4000 meshes per sq.cm. C.O.C.

Washing or Bleaching Nylon
Deutsche Gold- und Silber-Scheideanstalt *BP* 784,911

When bleaching nylon with per compounds degradation of the fibres is avoided by presence in the bath of an organic compound containing at least one N atom linked to at least one H atom or the oxidation product of such a compound, e.g. methylene diamine or *N*-hydroxy *N'*-phenyl urea. C.O.C.

Bleaching with Permonosulphuric Acid
Stevensons (Dyers) *BP* 784,437

Modification of *BP* 727,419 (J.S.D.C., **71**, 362 (1955)). The goods are treated at 80–100°C. with an aqueous solution of permonosulphuric acid. It is preferable that H_2O_2 should be absent but small amounts may be present provided that the concentration of permonosulphuric acid is maintained during the bleaching. The process is applicable to all fibres except wool. C.O.C.

Chemistry of Cellulose Degradation (XI p. 109)

VIII— DYEING

Applications of Colour Physics to Textiles
J. C. Guthrie and P. H. Oliver

J.S.D.C., **73**, 533–542 (1957)

Both additive and subtractive mixing of colours are discussed, leading to the C.I.E. system of colour specification, which is based on three primary colours and additive mixing. Measuring instruments and some types of illuminants are considered. Applications include examples of metameric matches, dichroism, quality control, and some problems in the blending of coloured fibres. AUTHORS

Hydrogen-bond Formation. VI— Rôle of Hydrogen Bonds in Dyeing Processes
D. S. E. Campbell, D. Cathcart, and C. H. Giles

J.S.D.C., **73**, 546–553 (1957)

The results of a refractometric study of the formation of intermolecular hydrogen-bond complexes in water between aromatic compounds, including sulphonated dyes, and a variety of other reagents, including casein, used as models of fibres, are interpreted as an indication of the nature of possible hydrogen-bond complexes between dyes and fibres. Some conclusions are— (*a*) Non-ionic polar groups in dyes and other aromatic compounds, whether sulphonated or not, can form intermolecular hydrogen-bond complexes in water with a variety of organic compounds. (*b*) Hydrogen-bond formation between dyes and fibres in presence of water appears to be possible with protein fibres, nylon, cellulose acetates, Terylene, and acrylonitrile fibres, but is less likely to occur with cellulosic fibres. Suggestions are made regarding the nature and the position of the dye-fibre bonds. AUTHORS

Polymerisation of Dyes in Solution
N. Mataga

Bull. Chem. Soc. Japan, **30**, 375–379 (June 1957)

The absorption spectra of soln. of 2:8-diaminoacridine (I) and 2:8-bisdimethylaminoacridine (II) have been measured in pyridine, ethanol, acrylonitrile, acetone, butanol, and water over a range of solute concn. In each case a well defined isosbestic point is obtained, showing that only two species, viz. dye and polymer $(dye)_n$ are present. Analysis of the bands of longest wavelengths enables n and the equilibrium constant of association—

$$K = \frac{x}{nc_0^{n-1}(1-x)^n}$$

(where $(1-x)$ = fraction of monomer and c_0 = analytical concn. of dye) to be calculated. For II $n = 2$ for all solvents at room temp., but for I $n = 2$ only in H_2O and C_2H_5OH; $n = 4$ in CH_2:CH·CN, CH_3·CO·CH_3 and C_4H_9OH; and $n = 8$ in C_5H_5N. The values of K bear no relation to the refractive index, dielectric constant, or dipole moment of the solvent. A.J.

Crystal Symmetry and the Adsorption of Dyes by Growing Crystals
J. Whetstone

II— Potassium Nitrate

J.C.S., 4284–4289 (Oct. 1957)

The suitability, for adsorption of dyes, of the principal low-index planes of potassium nitrate is discussed in the light of the previously proposed scheme (cf. *J.C.S.*, 4841 (1956)) to explain modifications of crystal habit by dyes. Evidence obtained from crystals of potassium nitrate modified by the dyes— Fast Red E (C.I. Acid Red 13), Amaranth (C.I. Acid Red 27), "D.I.F.", Solochrome Yellow YS (C.I. Mordant Yellow 20), disulphonated Döbner's violet, Ink Blue (C.I. Acid Blue 93), 1:4-diaminoanthraquinone-2-sulphonate, and "Acid Alizarin Blue" (C.I. Mordant Blue 23)— indicates that dye absorption influences not only formation of pleochroic dye inclusions but also twinning tendencies, owing to the pseudohexagonal symmetry of the crystals about the axis normal to {001}.

III— Sodium Nitrate and Ammonium Sulphate

Ibid., 4289–4294

The modifications of crystal habit produced in sodium nitrate and ammonium sulphate by 23 dyes are described. The lack of pleochroic dye inclusions observed in the modified crystals is explicable by their respective trigonal and pseudohexagonal symmetry, with dye adsorptions on planes perpendicular or steeply inclined to their modified basal planes. H.H.H.

Formation of Micelles of some Azo Dyes
H. P. Frank

J. Colloid Sci., **12**, 480–495 (Oct. 1957)

Solutions of Orange II (C.I. Acid Orange 7) and Benzopurpurin 4B (C.I. Direct Red 2) are studied by conductance, viscosity, and light-scattering measurements. Micellar particles of large size are formed only in the presence of added salt. The degree of association of Benzopurpurin is much greater than that of Orange II. It is suggested that the micelles represent rod-like particles and that the dye ions are associated in the manner of a stack of coins. A particle weight of 1,900,000 is obtained for Benzopurpurin in 0·04 M-KCl at 25°C. The particle weight of Orange II varies with concentration and temperature and reaches a value of 37,000 in 0·4 M-KCl at 5°C. Benzopurpurin forms micelles in the absence of salt if the pH approaches the isoelectric point. W.R.M.

Monolayers. VI— Reactions in Surface Films of Acetates and Ketones, and their Relation to Adsorption by Cellulose Acetate
A. Cameron, C. H. Giles, and T. H. MacEwan

J.C.S., 4304–4311 (Oct. 1957)

The surface films examined were monolayers of cetyl acetate, cellulose triacetate, and methyl and ethyl stearyl ketones, spread on water and aq. soln. of ethylene glycol, mesaconic acid, urea, ethyl methyl ketone, benzenesulphonic acid, benzoquinone, phenol, pyridine, quinol, sucrose, Acid Magenta, pinacyanol, 2:2′-dipyridyl, sulphato-ester dye, and anthraquinone-2-sulphonic acid; also a mixed film of cellulose triacetate with the non-ionic Dispersol Fast Crimson B for cellulose acetate. The mol.

area and compressibility data indicate that in presence of water the ketone group has much weaker hydrogen-bonding power than the acetate group, and the films of the ketones are consequently expanded much less by all the solutes than are the acetate films. Highly ionised cpd., including sulphonated dyes, cause considerable expansion of the cellulose triacetate films, even though they have no affinity for the same material in bulk. The results are used to interpret the mechanism of adsorption of solutes by cellulose acetates, where penetration into regions not normally swollen by water has some resemblance to solid soln. H.H.H.

Interaction of Polyvinylpyrrolidine with some Azo Dyes
H. P. Frank, S. Barkin, and F. R. Eirich
J. Physical Chem., **61**, 1375–1380 (Oct. 1957)

Polyvinylpyrrolidine shows strong affinity for Orange II (C.I. Acid Orange 7) and Benzopurpurin (C.I. Direct Red 2). One dye ion is bound by chain segments of 7 and 10 monomer units for Orange II and Benzopurpurin respectively. It is believed that the dye ions align themselves with their long axes along the polymer chain and are held in place by van der Waals forces. W.R.M.

Continuous Piece Dyeing
H. Waibel
Tint. ind., (27), 177–200 (May 1957)

A substantial review of the main methods, including a discussion of the advantages of continuous treatment and covering vat-acid, leuco-compound, and pigment applications. The pad–steam, Standfast, normal Williams, hot-oil and steam–liquid booster processes are described and compared. S.R.C.

Vivid Greens based on Phtalogen Dyes
H. Thaler
Melliand Textilber., **38**, 1038–1039 (Sept. 1957)

Sky blues and bluish greens only can be obtained with the existing range of Phtalogen (FBy) dyes. Interesting bright yellowish greens, of good all-round fastness, are achieved by topping Phtalogen-dyed material with selected azoic combinations, examples of which are given. L.A.T.

Increased Diffusion of Dyes into Cellulose
V. E. Rostovtsev
Tekstil. prom., **17**, 39–41 (June 1957)

The effects of salt concn. (10–30 g./litre) and ageing temp. (100–130°c.) on bleached, mercerised satin which has been impregnated with Direct Crimson are studied. Diffusion of the dye is found to increase at high temp. and high salt concn.; thus at 130°c. and 20–30 min. ageing time satisfactory dyeing is obtained. In the continuous dyeing of cotton fabric with black sulphur dyes, steaming increases the fastness to rubbing. G.J.K.

Continuous Dyeing of Cellulosic Fibres
H. Heuberger
Melliand. Textilber, **37**, 1427–1432 (Dec. 1956): *Teintex*, **22**, 745–757 (Oct. 1957)

Overdrying prior to dyeing must be avoided, and careful preparation to eliminate resists to the rapid dyeing process is necessary. A back-wetting agent is frequently advisable. Due to exhaustion in padding, light shades are more prone to tailing than heavy shades. Hot padding is necessary with the latter for greater dye solubility but lower temperatures with light shades can be used to reduce strike. Concentrated dye standardisations are preferable to reduce salt exhaustion effects. Dye solubility can often be improved with pyridine-based assistants. The expression of a pad depends on the pressure between and the diameter of the rollers, the hardness and thickness of the covering, the temperature of the baths, the cloth speed, the presence of e.g. thickening agents and the nature of the goods. Small rollers thinly covered with hard material give low expression, as does reduction of viscosity with rise in temperature, and high cloth speed. The pad–salt, pad–steam, and Pad–Roll processes are compared. S.R.C.

Artificial Ageing of Cotton Fabrics dyed with Sulphur Black
Z. I. Sergeeva and I. L. Khmel'nitskaya
Tekstil. prom., **17**, 42–44 (June 1957)

Tendering of cotton dyed with sulphur blacks is studied on different samples of madapolam dyed with Sulphur Black K (15% on the wt. of material), Indocarbon TsL (15%), and Direct Black ZS (4%) respectively. The samples are submitted to ageing for periods of up to 20 hr. in a special steam ager (temp. 130–135°c.; amount of water fed 75–80 ml./hr.; threefold change of air per hour), and changes in copper number and pH are measured. Tendering is greatest in the case of Sulphur Black K, and only slight in the case of Direct Black ZS under the above conditions. G.J.K.

Automation in Vat Dyeing
I. D. Zakhar'evskaya, V. A. Lentovskaya, and M. S. Zakhar'evskiĭ
Tekstil. prom., **17**, 37–38 (June 1957)

Advantages of automation in vat dyeing are discussed. A relatively simple scheme based on the control of the redox potential and concn. of each dye is put forward, and preliminary experiments on Red KKh, Golden Yellow ZhKh, Blue O, and Bright Green Zh vat dyes are described. G.J.K.

Levelling Problems in Vat Dyeing
H. Musshoff
J.S.D.C., **73**, 543–546 (1957)

An important factor in the poor levelling of certain vat dyes is slow migration due to molecular aggregation (up to 3000 molecules) of the leuco compound in solution. Migration may be improved by addition to the dyebath of a suitable non-ionic solvent, e.g. Solidegal GL (CFM). Practical methods for overcoming levelling difficulties in package dyeing and piece dyeing, notably the vat-acid and pad–steam processes, are discussed. AUTHOR

Dyes for Cellulose Acetate and Synthetic Fibres. I— 1:5- and 1:8-Acylamino-hydroxyanthraquinones
L. M. Egorova, V. N. Ufimtsev, and E. S. Yampol'skaya
J. Gen. Chem. U.S.S.R., **27**, 1650–1654 (June 1957)

The absorption spectra, solubility in ethyl acetate, and substantivity on cellulose acetate of 1-acylamino-5- and -8-hydroxyanthraquinone are investigated. Increasing size of the acyl group causes a marked decrease in substantivity, but this decrease cannot be correlated with the solubility in ethyl acetate. G.J.K.

Modification of Wool Serge during Piece Dyeing
P. Outreman, G. Mazingue, and M. van Overbèke
Bull. Inst. Text. France, (66), 41–51 (April 1957)

Measurement of the bursting strengths of wool fabrics before and after commercial piece dyeing shows a definite reduction in mean bursting strength after dyeing. Measurements on samples dyed in the laboratory at various pH values show that damage is a minimum in the isoelectric region (pH 3·5–4·5). Further laboratory work indicates that severe damage can occur when dyeing with 1:1 metal-complex dyes in presence of H_2SO_4 without the addition of neutral salt. Alkali solubility, bursting strength, and abrasion test data are given. J.C.F.

Sorption of Monobasic Dye Acid by 6-Nylon Fibre
M. Nishio
Bull. Chem. Soc. Japan, **30**, 363–367 (June 1957)

pH isotherms for the equilibrium uptake of the purified dye acids of Orange II (C.I. Acid Orange 7) and Metanil Yellow YK (C.I. Acid Yellow 36) on 6-nylon (polycaproamide) have the characteristic shape of isotherms for 6,6-nylon. Equilibrium uptake is constant over the pH range ~ 4–6 at a value of ca. 0·05 equiv./kg., corresponding to a terminal $-NH_2$ group analysis of 0·043 equiv./kg. Affinities, calculated by the Gilbert–Rideal method, are −15·7 kcal./mole for Orange II dye acid and −17·8 kcal. per mole for Metanil Yellow YK, the respective dye anion affinities being −3·8 and −5·9 kcal./mole. A.J.

Continuous Dyeing of Fabrics of Polyamide and Polyester Fibres
A. Würz
Melliand Textilber., **38**, 777–783 (July 1957)

Two padding processes are described for the application of metal-complex dyes on fabrics made from continuous-filament polyamide yarns. In the first method, the padding liquor consists of a water-in-oil emulsion of the dye, and in the second process the dye is dissolved in an organic solvent with subsequent addition of a thickening agent. After preliminary drying, the colour is fixed by steaming, dry heat, or treatment with dilute formic acid. In this way, it is possible to obtain fast dyeings without the risk of barriness due to physical variations in the yarns. The

emulsion padding method was found useful also for the production of dark colours on polyester fibres.

L.A.T.

Dyeing of Acrylic Fibres

F. Schouteden

Melliand Textilber., **38**, 65–68 (Jan. 1957)

In the proposed method for dyeing acrylic fibres, hydroxylamine sulphate is used. Thus 100 g. of fibre is treated for 4 hr. at the boil in a solution containing 5·0 g. hydroxylamine sulphate and 1·2 g. sodium hydroxide, at a liquor ratio of 20:1. Under these conditions it is estimated that about 2·5% of available nitrile groups have reacted with hydroxylamine. The principal reaction is probably formation of amidoxime—

$$-C{\equiv}N + NH_2{\cdot}OH \rightarrow -C(=N{\cdot}OH)(NH_2)$$

The above treatment produces some yellowing, but the material can be cleared by boiling in 0·1 N-H_2SO_4. Tensile strength is virtually unaffected, although the loop strength of the treated fibres is slightly lowered. The uptake of disperse, direct, metal-complex, and particularly acid dyes is considerably enhanced.

L.A.T.

Mechanism of the Dyeing of Acrylic Fibres with Basic Dyes—I and II

O. Glenz and W. Beckmann

Melliand Textilber., **38**, 296–300 (March); 783–787 (July 1957)

I— Dyeing equilibria and surface (zeta) potentials have been investigated. On the basis of adsorption isotherms and analytical data the presence of active acidic groups in acrylic fibres is postulated. It is suggested that the dye cations are first adsorbed on the negatively charged fibre surface and subsequently diffuse into the fibre. Then they combine with the active acidic groups. Their accessibility depends on the temperature and the state of the fibre. Saturation values have been determined for a number of dyes, and a procedure is described for practical application of the results.

II— Potentiometric titrations as well as reaction with Malachite Green and Carbinol base allow quantitative estimation of acid groups in the polyacrylonitrile fibres to be made. The estimated value of $3{\cdot}5 \times 10^{-5}$ g.-equiv./g. fibre is in fairly good agreement with the average molecular weight value determined by the viscometric method. Experimental work is described which led to a formula for the rate of dyeing based on the dye concentration in the fibre and in solution, saturation value, and zeta potential. The rate of dyeing increases exponentially with temperature.

L.A.T.

Chemistry and Technique of Dyeing Acrylic Fibres by the Cuprous Ion Method

H. Rath, H. Rehm, H. Rummler, and E. Specht

Melliand Textilber., **38**, 431–435 (April); 538–542 (May 1957)

Several reducing agents have been investigated and particular reference is made to ferrous sulphate and sodium sulphite. Electrolytic reduction of cupric ions in presence of metallic copper has also been studied.

L.A.T.

PATENTS

Colouring Warps during Weaving

P. Stevens *BP* 783,134

Behind the loom there are a series of dyetanks into which the threads of the warps are dipped in accordance with the movements of shedding apparatus. This enables e.g. a single thread to be treated with 5 different colours in different places. This enables 80% decrease in the number of threads required for a carpet woven in five colours.

C.O.C.

High-temperature Dyeing

Fryman & Fletcher *BP* 784,394

The goods are impregnated with an aqueous solution or dispersion of dyes which are unaffected at 160–235°c. The impregnated goods are then treated in air at 160–235°C. in absence of water vapour other than that liberated from the goods, long enough to dry the goods and fix the dyeings. Thus cloth containing Terylene, wool and viscose rayon was impregnated with an aqueous liquor containing Serisol Yellow 5GD (YDC), C.I. Acid Red 73, Durazol Fast Blue 3RS (ICI), NH_4 thiocyanate, NaCl and Na lauryl sulphate. It was then passed down a vertical tube. The tube was surrounded by heating jackets so arranged that the first zone of the tube is at 210°c. and the bottom zone at 180°c. There was the minimum movement of air through the tube the bottom of which was sealed with cold water. The cloth passed through the tube at 4 ft./min. to allow the goods to become dry while in the 210°c. zone. This dyed the Terylene yellow, the wool red and the viscose rayon blue.

C.O.C.

Dyeing Peroxide-bleached Wool

Food Machinery & Chemical Corpn. *BP* 784,205

Treating peroxide-bleached wool with such small amounts of reducing agents that they have no bleaching action results in subsequent acid dyeing of the wool being of improved hue and colour value with reduction in variation between the dyeings of different batches.

C.O.C.

Dyeing Wool with Metal-complex Dyes

FBy *BP* 784,664

When dyeing wool with metal-complex dyes containing < 1 atom of a metal per molecule of dye which may contain a carboxyl group participating in the complex formation but no other acid, water-solubilising group, very level dyeings are obtained at pH 4–5 by using agents produced by the action of > 29 mol. of ethylene oxide or 1 mol. of a compound containing a hydroxyl group or a primary or secondary amino group of > 2 C, e.g. stearyl or oleyl alcohol or cetylamine.

C.O.C.

Fast Dyeings and Prints on Organic Derivatives of Cellulose

BASF *BP* 782,785

Very fast dyeings and prints are obtained on cellulose acetate, cellulose triacetate and ethyl cellulose with dyes containing a carboxylic acid, a sulphonic acid or sulphonic acid amide group or two or more of these groups which may be the same or different by treating the cellulose derivative before or during dyeing with a carbamic acid ester of a compound containing at least one hydroxyl group and at least one tertiary amino group. Thus acetate rayon is treated for 30 min. at 75–80°c. with an aqueous dispersion of the carbamic acid ester from 1 mol. of phenyl*iso*cyanate and 1 mol. of 1:3-bis-diethylamino*iso*propanol, rinsed and dyed for 30 min. at 80°c. with the dye 1-amino-4-(*m*-cyanophenyl)-aminoanthraquinone-2-sulphonic acid from a sulphuric acid bath. This yields deep blue dyeings of very good fastness to light and good–very good wet fastness properties.

C.O.C.

Dyeing Mixtures of Acrylic and Cellulose Derivative Fibres

Société Rhodiaceta *BP* 784,191

The cellulose derivative component is first treated, or has incorporated in it, a colourless organic nitrogenous compound having affinity for it and the mixed fibres are simultaneously or subsequently dyed by the cuprous ion process. Thus fabric containing polyacrylic fibres and acetate rayon is treated with a 10% aqueous solution of the formaldehyde-bisulphite complex of bis-4:4′-(cyanoethylamino)-di*cyclo*hexylamine and then dyed with a mixture of C.I. Acid Blue 25 and C.I. Acid Red 88 for an hour in an acetic acid bath after which $CuSO_4$ and sodium formaldehydesulphoxylate are added and dyeing continued for 30 min. This yields a very level royal blue dyeing.

C.O.C.

Dyeing and Printing Textiles and Foils of Organic Esters

S *BP* 784,668

Dyeings and prints fast to light and washing are obtained on acetate rayon, cellulose triacetate, Terylene etc. by applying anthraquinone acid dyes containing a sulphonic acid group in the 2-position as the only solubilising group and then dyeing or steaming the print in presence of a reducing agent which splits off the sulphonic acid group from the dye. Thus Terylene is entered at 60°c. with the dye Na 3′-carbethoxy-4-(2′-cyano)-propylamino-1:9-anthrapyridine-2-sulphonate in a bath containing Zn formaldehyde-sulphoxylate or 2-hydroxy-1:1′-diphenyl as swelling agent and the bath brought to the boil in 20 min. and dyeing continued at this temperature for 60 min. This yields a level yellow dyeing of good fastness to light and washing.

C.O.C.

Dyeing Polyacrylonitrile Fibres
Monsanto *USP* 2,746,836

Polyacrylonitrile fibres can be dyed with direct dyes in presence of a quaternary phosphonium salt. Thus a yarn of an acrylonitrile-vinylchloride (9:1) copolymer is satisfactorily dyed with C.I. Direct Yellow 11 if 8% of benzyl tris(dimethylamino) phosphonium bromide is present in the dyebath. C.O.C.

Pigmented Resins for Pad-dyeing or Printing
Interchemical Corpn. *USP* 2,744,035

A dispersion of a pigment in an aqueous solution of a resinous polyamine acetate and a polyacryl compound of pH < 6 is especially suitable for padding into textiles followed by heating to cross link the polyamine with the polyacryl compound. C.O.C.

Adding Sulphur Dyes to Viscose Spinning Solutions (VI p. 103)
Practical Causes of the Chemical Degradation of Rayon and Properties of Modified Celluloses (VII p. 104)

IX— PRINTING

Rongalit H— A New Reducing Agent for the Discharge Printing of Vat Dyes on Cellulose Acetate
G. Bernardy and H. Wirth

Melliand Textilber., **38**, 1044–1049 (Sept. 1957)

In contrast to existing methods of discharge printing, which are favoured by acidic conditions, Rongalit H (BASF) is applied under slightly alkaline conditions in presence of triethanolamine, without the risk of local saponification of cellulose acetate. This method is applicable not only to a large number of vat dyes but also to selected disperse dyes. Promising results were also obtained with Rongalit H in both the direct and discharge styles of printing on cotton, cellulose triacetate, and viscose rayon. L.A.T.

PATENTS

Embossed Prints
Ciba *BP* 784,336

Embossed prints are obtained on materials treated with a resin precondensate by applying with a hot embossing roller a pigment paste containing a metal drier and a resin which undergoes oxidative drying in presence of the drier and contains > 3 unsaturated groups per molecule, e.g. an alkyl ether of a polymethyl compound containing > 3 alkyl ether groups. This gives speedy printing of depressed portions of the patterns and yields prints of considerable all round fastness. C.O.C.

Pigment Prints with the aid of a Styrene–Butadiene Copolymer Binder
Chemische Werke Hüls *BP* 784,880

When using a copolymer of 50–90% styrene and 10–50% butadiene as the binding agent in pigment printing, prints which on staple fibre withstand prolonged and repeated washing at 60–80°C. while being rubbed are obtained if 0·5–5·0% of an ammonium salt, e.g. NH_4 thiocyanate, is added to the printing paste. C.O.C.

Pigment Emulsions for Printing and Padding
FBy *BP* 784,946

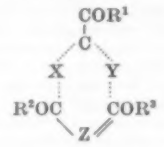
Modification of *BP* 729,122 (J.S.D.C., **71**, 426 (1955)). Basic high mol. wt. compounds containing amino and/or imino groups and reactive epoxy groups, e.g. the condensate obtained by treating di- or polyamine with an epihalogenohydrin or dihalogenohydrin, is used to obtain cross-linking of the high molecular basic compound. Printing pastes containing such cross-linking agents are viscous and so do not require any thickening agent to be present, this in turn results in prints which are faster to washing and of better handle. C.O.C.

Application of Transfers
Cromtryck *BP* 784,966

A transfer made up of a backing united to an image layer by a water-soluble adhesive. The image layer has a smooth non-tacky surface and includes a constituent which on heating softens and becomes an adhesive. It is soaked in water and applied without heat and pressure to the surface to be decorated, the backing removed, the transferred layer allowed to dry and the whole heated to 80–200°C. to effect adhesion of the image layer to the surface to be decorated. Such a transfer is particularly useful for application to articles to be oven dried. C.O.C.

Colour Photography
Kodak *BP* 784,479

A reducible silver salt image is treated with a primary aromatic amino developer in presence of a colour coupler which forms an insoluble dye and a "competing" coupler which forms a soluble dye which later is washed out of the emulsion. The competing coupler competes with the colour coupler for the oxidised developer and so permits formation during colour development of oxidised developer in excess of that needed to produce dye images of normal contrast. This, in turn, permits use of larger amounts of silver halide in the emulsion and so results in dye images of normal contrast without the exaggerated graininess and low sharpness and definition of those obtained by normal procedures. The competing coupler has the formula—



(R^1 = OH, alkoxy or amino; R^2 and R^3 = H or acyl; X and Y = atoms to complete a 6-membered ring; Z = N or CH), e.g. 2:6-dihydroxy*iso*nicotinic acid. C.O.C.

Pre-sensitised, Positive-working Offset Diazotype Printing Plate
General Aniline *BP* 784,001

Colouring Warps during Weaving (VIII p. 106)
Fast Dyeings and Prints on Organic Derivatives of Cellulose (VIII p. 106)
Dyeing and Printing Textiles and Foils of Organic Esters (VIII p. 106)
Pigmented Resins for Pad-dyeing or Printing (VIII this page)

X— SIZING AND FINISHING

Weathering Characteristics of Cotton Fabrics containing Inorganic Compounds. II— Further Work on Copper–Chromium Treatments
G. R. F. Rose and C. H. Bayley

Text. Research J., **27**, 519–528 (July 1957)

The study was made on unbleached and bleached cotton duck treated with (*a*) copper carbonate and chromic oxide, (*b*) chromic oxide aftertreated with copper naphthenate and with copper 8-hydroxyquinolinolate, and (*c*) chromium naphthenate mixed with copper naphthenate and copper 8-hydroxyquinolinolate respectively; and unbleached cotton sheeting treated with a mixture of copper carbonate and chromic oxide. The treatments were applied at two levels of copper for each compound and the chromium added so as to give ratios of Cr_2O_3:CuO at, above and below unity. The optimum ratio of around unity found in the earlier work was confirmed for all the above mixtures. Mixtures of copper and chromium in inorganic form appear to afford greater protection than organic or organic–inorganic mixtures. The presence of noncellulosic materials enhances the protective effect of the above mixtures as does also an increase in fabric weight. S.B.D.

Improvement of the Creasing and Swelling Properties of Viscose Rayon Fibres
A. Geiger

Textil-Rund., **12**, 494–502 (Sept.); 560–570 (Oct. 1957)

The effects of— (*a*) polymers of, or styrene copolymers of, hydroxymethyl vinyl ketone and α-hydroxymethyl-vinyl methyl ketone; (*b*) mono- and polyfunctional chloro compounds (e.g. bischloromethyl-*m*- and -*p*-xylol, phosphonitrile chlorides, and cyanuric chloride), and (*c*) *p*-toluene-sulph*iso*cyanate were studied. S.R.C.

Preparation and Properties of Regenerated Cellulose containing Vinyl Polymers. III— Moisture Relations
G. Landells, J. A. Leathley, and C. S. Whewell

J.S.D.C., **73**, 496–500 (Nov. 1957)

Large quantities of hydrophobic vinyl polymers deposited inside viscose rayon fibres reduce water imbibition comparatively slightly, but hydrophilic polymers increase the imbibition. Measurements of the

absorption–desorption isotherms of fibres containing various polymers show that deposition of polymethyl methacrylate or polystyrene produces little effect on the moisture contents calculated on the weight of cellulose. Introduction of hydrophilic polymers increases the moisture contents of the fibres. AUTHORS

Mechanism of the Reaction between Cystine in Keratin and Sulphite–Bisulphite Solutions at 50°C.—I
P. T. Speakman
Biochim. Biophys. Acta, **25**, 347–355 (Aug. 1957)

The results of setting experiments show that SO_3^{2-} and OH^- are the main reactive ions when the cystine disulphide cross-linkages in human hair are treated with sulphite–bisulphite solutions in 10% ethanol at 50°C. *S*-Cysteine sulphonate, $RSSO_3^-$, incorporated in the protein chain, is the most important source of bisulphite-stable set at the lower pH values (4–8) investigated. The cross-linkages involved are formed in the reaction mixtures at 50°C. and also when a treated fibre is immersed free from tension in boiling distilled water. The cross-linking is alkali-catalysed. The sulphenic acid group, RSOH, incorporated in the protein chain also gives rise to bisulphite-stable cross-linkages; its effect becomes appreciable at pH values above 8. Together with other evidence, this suggests that the cross-linkages in this case are lanthionine, RSR, bonds. P.G.M.

Formation of Disulphides during Hydrolysis of Proteins containing Oxidised Thioether Groups
J. M. Swan and E. F. Woods
Biochim. Biophys. Acta, **25**, 432–433 (Aug. 1957)

Various wool proteins, prepared by treating wool with iodoacetic acid or iodoacetamide and subsequently oxidising with peracetic acid, were shown on hydrolysis to give an apparent high cystine content. It is suggested that this is due to the decomposition of sulphoxide or sulphone during hydrolysis to yield a new disulphide. P.G.M.

Chemical Anti-felting Reaction for Wool
C. Earland
Text. Research J., **27**, 587–588 (July 1957)

Comment is made on the ability of several powerful oxidising agents to prevent felting in wool fabrics, this being compared with their effect on silk fibroin. S.B.D.

Yellowing of Nylon
F. H. Steiger
Text. Research J., **27**, 459–465 (June 1957)

Treatment with thermosetting formaldehyde resins increases the tendency of nylon to yellow when heated. The terminal amine groups are involved in the discoloration, but these can be blocked by reaction with urea under normal textile curing conditions to give a fabric with less tendency to yellow. S.B.D.

PATENTS

Durable Lustrous Finish
Joseph Bancroft & Sons Co. *BP* 784,764

Fabric impregnated with the precondensate of a resin is fed to a carrier blanket which carries the fabric against and away from a drying drum. Suitable adjustment of the temperature of the drum and the contact pressure of the fabric enable the degree of lustre to be controlled. Finally the fabric is baked to set the resin. C.O.C.

Reducing the Felting Power of Wool
Wolsey *BP* 783,126

When treating wool with chlorinating and brominating agents more level results and a better handle are obtained if the agents are added as a drip feed so that each 1% (on wt. of goods) of Cl or Br added takes 5–10 min. to add. C.O.C.

Water-proof Non-flammable Textiles
BrC *BP* 784,318

Cellulose or cellulose acetate yarn is coated with 80–150% of its weight of a rubber-like, water-resistance, non-flammable, film-forming material, e.g. plasticised poly(vinyl chloride). It is then formed into fabric and hot pressed to bind the yarns together by coalescing their coatings. This yields a product which is much more flexible and of higher strength–weight ratio than is a coated fabric of similar waterproof and nonflammable properties. C.O.C.

Flame-resistant and Water-repellent Finish
K. Quehl *BP* 784,171

The materials are treated with an aqueous solution or dispersion containing a water-repellent agent, a chlorinated organic compound having a fixative action, an organic acid and stabiliser, an Al or Zr salt and a metal oxide having flame protecting properties. Thus an aqueous dispersion of paraffin wax and Al formate rendered acid with formic acid and stabilised with glue had stirred into it a 70% chlorinated paraffin wax and a little pentachlorophenol dissolved in a mixture of trichloro- and perchloro-ethylene. Finally there was added antimony trioxide and a pigment. Canvas impregnated with this dispersion was coloured and had very good resistance to water and rotting as well as excellent resistance to flame. C.O.C.

Antistatic Finish for Synthetic Fibres
ICI *BP* 784,052

The fibres are treated with a polymeric quaternary ammonium compound whose monomer is of formula—

$$CH_2{:}C(R^1)COOR^2N^+(tert.)X^-$$

(R^1 = H or CH_3; R^2 = alkylene in which the chain may be interrupted by heteroatoms; N(*tert.*) = an aliphatic or heterocyclic tertiary amine; X = monovalent amine) e.g. quaternised poly(diethylaminoethylmethacrylate). The anti-static finish obtained is very resistant to washing in aqueous soap or soda. C.O.C.

Nylon Finish for Increasing the Moisture Absorbing Properties of Nylon Fabrics
Hans C. Bick *USP* 2,746,941

Nylon fabric is rendered moisture-absorbent by treating it with an aqueous emulsion of nylon, e.g. an emulsion made up of Type 8 nylon (DuP), sucrose octa-acetate, Tergitol Dispersant NPG, alcohol, and water. C.O.C.

Flexible, Vapour-permeable, Water-resistant Thermoplastic Coated Fabric
B. F. Goodrich Co. *BP* 782,719

Fabric is coated with a polymer of a halogenoethylene having 1 or 2 Cl, Br or F atoms on only one C atom in which has been incorporated 50–300% by wt. of the polymer of a powdered non-hygroscopic material soluble in a non-solvent for the polymer. It is then treated with such a solvent to remove the powdered material. The product is permeable to water vapour but not to liquid water. Such a composition can also be made into a self-supporting film and similarly treated. C.O.C.

Finishing Plastic Sheets or Coatings
J. H. Benecke *BP* 782,522

Thermally decomposable salts are applied in the solid form to the surface of the plastic while it is soft, the plastic then hardened and heated to decompose the salts. Thus cotton sheeting is coated with poly(vinyl chloride), with Cd stearate as stabiliser, dioctyl phthalate as plasticiser and C.I. Pigment Green 17 as the colorant. Ammonium carbonate of particle size 0·1–2·0 mm. is then sprinkled on the surface, the coating allowed to harden and then heated to drive off the ammonium carbonate. The finished sheeting has a grained appearance. C.O.C.

Artificial Deerskin
Deutsche Gold- & Silber-Scheideanstalt *BP* 784,190

A substrate is coated with a thin layer of a coloured poly(vinyl chloride) paste containing a plasticiser. The coating is then sprayed with a powder which volatilises at 100–200°C. after which it is heated to volatilise the powder and complete gelation of the poly(vinyl chloride). C.O.C.

Rendering Glass Fibre Fabrics Readily Cleansable
Owens-Corning Fiberglas Corpn. *USP* 2,744,835

The fabrics are first treated with an aqueous composition containing a vinyl methyl ether–maleic anhydride copolymer and a vinyl copolymer, e.g. Rezosol 550 (E. F. Houghton Co.), then baked and finally treated with a Werner complex in which the acido group coordinated with the trivalent nuclear Cr atom contains > 9 C atoms. C.O.C.

Hiding Power of Titanium Pigments (V p. 101)
Comparative Study of the Behaviour at High Temperatures of Partially Acetylated Cotton and some other Fibres (VI p. 102)
Structure of Cellulose by Alcoholysis (XIV p. 114)

XI— PAPER AND OTHER CELLULOSIC PRODUCTS

Weak Points in the Chains of Natural Cellulose Fibres

A. Parisot and G. Champetier

Chim. et Ind., **77**, 1317–1324 (June 1957)

The assumption of weak points in the cellulose chain, particularly sensitive to breakage, is still controversial. Mechanical properties of dry and wet fibres, after various washing and bleaching treatments, are considered in terms of degradation. Mechanical resistance of wet fibres is generally greater than that of dry fibres, but chemical resistance is less. This can be explained by the existence of regularly spaced weak points. If so, there should be greater accessibility of water and reagents at these points. The mechanical resistance of wet fibres is equal to that of dry fibres at a D.P. of about 600, suggesting that weak points may be separated by about 600 glucose units. Chromatography of fibre hydrolysates at various stages of degradation does not permit any explicit conclusion as to the nature of the weak points. W.R.M.

Dissolution of Cellulose in Quaternary Ammonium Bases

A. A. Strepikheev, I. L. Knunyants, N. S. Nikolaeva, and E. M. Mogilevskiĭ

Izvestiya Akad. Nauk S.S.S.R., otdel. khim. nauk, (6), 750–753 (June 1957)

Complete dissolution of cellulose is found to take place only in dimethylphenylbenzyl, triethylbenzyl, and triethylfurfuryl quaternary ammonium hydroxides. A mechanism for the dissolution of cellulose is suggested. G.J.K.

Chemistry of Cellulose Degradation

P. Rochas

Bull. Inst. Text. France (65), 15–40 (Feb. 1957)

The subject is reviewed under the following headings— (i) chemical structure of cellulose; (ii) (*a*) degradation to hydrocelluloses by acid, (*b*) oxidation of hydroxyl and aldehyde groups, (*c*) fission of oxycelluloses by alkalis; (iii) methods of identifying functional groups characteristic of modified celluloses. Particular attention is given to the work of the Lyons school. J.C.F.

Influence of the Ketonic Group on the Stability of the Glucosidic Linkage

Z. I. Kuznetsova, E. D. Kaverzneva, and V. I. Ivanov

Izvestiya Akad. Nauk S.S.S.R., otdel khim. nauk, (5), 655–656 (May 1957)

The influence of the ketonic group on the stability of the glucosidic linkage is studied by submitting monoacetyldihydroxyacetone tetra-acetyl-β-glucoside (I), glycerol tetra-acetyl-β-glucoside (II), and β-methylglucoside (III) to hydrolysis. The stability of I is compared with that of II and III and is found to be somewhat decreased in acid media and very strongly decreased in alkaline media. G.J.K.

PATENT

Maintenance of Brightness in Bleached Wood Pulp

Ford Machinery & Chemical Corpn. *BP* 785,737

A pulp which retains its brightness is obtained by the incorporation of hydrogen peroxide into the pulp just prior to baling, storage or shipment. The required amount of hydrogen peroxide is ·02 to ·025% based on the weight of the pulp. G.S.B.

Alkaline Degradation of Polysaccharides (VII p. 103)

Practical Causes of the Chemical Degradation of Rayon and Properties of Modified Celluloses (VII p. 104)

False Viscosity in Cellulose Acetate Solutions (XIV p. 114)

XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Collagen Reactivity with Metals

R. Borasky

J. Amer. Leather Chem. Assocn., **52**, 596–610 (Nov. 1957)

The effects of treating collagen with soln. of 13 different metal-containing cpd. have been studied by means of measurements of shrinkage temp. and electronmicroscopic appearance. Metals in the form of, or as part of, simple ions, do not combine with collagen. When the metal is part of an ionic complex it reacts with collagen, giving greater electron density and, usually, greater hydrothermal stability. Metal cpd. which are non-ionic but which react with –C:C–, terminal amino, terminal OH, and non-peptide-NH groups combine with collagen to give increased electron density and greater hydrothermal stability. The significance of these results in relation to the deposition of metals in skeletal tissue, and in explaining a mechanism of mineral tannage is discussed. J.W.D.

Reactive Groups of Collagen. II— Effect of the Reactive Groups on the Fixation of Certain Aldehydes

S. M. Bose and K. T. Joseph

J. Amer. Leather Chem. Assocn., **52**, 200–207 (April 1957)

The fixation of HCHO, acrolein, and crotonaldehyde by untreated collagen and several modified collagens (deaminated, deamidated, methylated, hypobromite-treated, acetylated, and benzoylated) and also by Ultramid 6A has been studied in soln. of acid, neutral, and alkaline pH. At acid and neutral pH the free amino and amide groups, at alkaline pH the guanidino groups, and at neutral and alkaline pH the OH groups take part in the reactions with aldehydes. It has also been shown that there is no primary reaction between the aldehydes and the peptide groups or the carboxyl groups. J.W.D.

Action of Nitrous Acid on Collagen

A. Küntzel

Leder, **8**, 34–38 (1957): *J. Amer. Leather Chem. Assocn.*, **52**, 645 (Nov. 1957)

Deamination of collagen and action of HNO_2 on amino acids are reviewed. Decreased swelling in acid of deaminised collagen has been cited as evidence that HNO_2 tans, but although leather does not swell, lack of swelling is not proof of tannage. The deaminised collagen does not show restricted swelling in alkali. Nitrite-treated skin will dry to a leather-like product, but if thoroughly washed after the nitrite treatment, it will dry hard and horny. Pretreatment of skin with nitrite before Cr tanning results in a softer, more leathery product, but the same effect can be obtained with nitrite in the pickle. Nitrite will mask Cr salts and increase the basicity of the solution, but the nitrito complex decomposes after some time, releasing HNO_2 and forming a high-basicity, unstable Cr compound. Softer leather is formed because nitrous gases mechanically open the fibres. Pretreating skins with nitrite before Cr tanning was of no practical value because of bad drawn grain; however, the process might be useful for chamois or chrome splits. C.J.W.H.

Cross-linking of Collagen with Aliphatic Epoxides

R. L. Sykes

J. Soc. Leather Trades Chem., **41**, 199–206 (June 1957)

The reaction between collagen and several aliphatic multifunctional epoxides has been studied. The application of the theory of rubber-like elasticity to the stress–strain characteristics of hydrothermally contracted collagen tendons before and after tannage indicates that the epoxides act as cross-linking agents. There is a direct relationship between the rise in the shrinkage temp. and the log. of the number of introduced cross-linkages; this may be a general relationship for all tannages where the bond between tanning agent and collagen is directional. Aliphatic epoxides produce a white leather requiring a min. of fat-liquoring but which is less strong than vegetable-tanned leather; this leather did not discolour or yellow appreciably after six months' exposure to a laboratory atmosphere. J.W.D.

Modern Finishes for Leather

W. O. Nutt

J. Soc. Leather Trades Chem., **41**, 189–199 (June 1957)

Resin binders of the acrylic and diene types commonly used in finishing are compared in the light of finish requirements, and the effect of components such as stabilisers (for diene resins) and emulsifying agents discussed. Other materials in common use such as nitrocellulose, casein, shellac, and HCHO fixes, are discussed in relation to the finish as a whole. The importance of the vol. of the pigment and other spacing material in a compounded film

is shown, illustrating the importance of the pigment–volume concn. requirements of different leathers. The preparation of leathers of different types, and further factors affecting the results obtained in resin finishing are detailed and comments given for the practical finisher. Tests applicable to resin binders and suitable for use in the tannery laboratory are described. J.W.D.

New Developments in the Opaque Coating Field— II
K. Eitel

Leder, **7**, 126–133 (1956):
J. Amer. Leather Chem. Assocn., **52**, 650 (Nov. 1957)

Developments in pasting, pigment finishes, and polymeric binders during the past five years are reviewed. C.J.W.H.

PATENT

Filling Leather with Modified Vinyl Ether–Maleic Anhydride Copolymers
General Aniline *USP* 2,746,837

Leather can be given body and fullness by treating it before, during or after tanning, with a vinyl ether–maleic anhydride copolymer in which 5–15% of the anhydride groups have been converted to the salt-half amide with ammonia or an aliphatic primary or secondary amine. They are particularly useful as adjuncts to chrome tanning. Their application causes no alteration in the affinity of the leather for dyes. C.O.C.

XIII— RUBBER; RESINS; PLASTICS

Aggregations of Charged Macromolecules
F. Oosawa

J. Polymer Sci., **26**, 29–45 (Oct. 1957)

The dispersion–aggregation equilibrium of charged macromolecules in solution is discussed. Aggregates may, under suitable conditions, be fibrous. The size and the shape of aggregates depends on the difference in chemical potential between macromolecules in the aggregated and disperse phases, the surface free energy per molecule in aggregates, and the magnitude and size of the long-range potential between macromolecules. States of aggregation are classified, and changes of state of aggregation under various conditions described. W.R.M.

Swelling and Solution of Synthetic Fibre-forming Polar Polymers in Liquids
W. R. Moore

J.S.D.C., **73**, 500–506 (Nov. 1957)

The general principles governing the interaction of organic liquids and synthetic fibre-forming polymers (including cellulose acetate) are considered, and the factors which determine whether a liquid will dissolve or swell a given polymer are discussed. AUTHOR

Isotactic Polymers
G. Natta

Chem. and Ind., 1520–1530 (23 Nov. 1957)

Isotactic, syndiotactic, and atactic polymers are distinguished and examples given. The crystallinity of isotactic polymers is briefly discussed. Stereospecific catalysts and the mechanism of polymerisation are considered. The reaction mechanism is of the anionic type. Processes controlling the molecular weights of polymers are considered. Atactic and block copolymers and the properties of mixtures of stereoisomers are discussed. Tables are given providing data on mechanical and physical properties of atactic, syndiotactic, and isotactic polymers. W.R.M.

Isotactic and Stereoisomeric Polymers
G. Natta

Chim. et Ind., **77**, 1009–1031 (May 1957)

New types of linear polymers, with regular steric structures, have been obtained by use of new stereospecific catalysts. These are crystalline isotactic polymers, in which tertiary carbon atoms in the main chain give the same relative steric arrangement, and syndiotactic polymers, in which each tertiary carbon atom gives the opposite steric arrangement to that of neighbouring atoms. Data are given on the crystalline structure and the properties of isotactic polymers of α-olefins, particularly poly*iso*propylene. Atactic polymers and polymers with "stereoblocks" have interesting elastomer properties. Stereo-specific polymerisation processes make it possible to produce new polymers including all the predicted stereoisomeric polymers of butadiene. W.R.M.

Homopolymerisation of *N-iso*Propylacrylamide
W. C. Wooten, R. B. Blanton, and H. W. Coover

J. Polymer Sci., **25**, 403–412 (Sept. 1957)

The rate of polymerisation of *N-iso*propylacrylamide in aqueous solutions, using a catalyst of ammonium persulphate and sodium bisulphite, is very sensitive to the pH of the system. A maximum rate is observed at pH 6·5. For a given catalyst concentration, the viscosity of the polymer is independent of rate when the pH is 6·5 or less. Above pH 6·5 the viscosity is inversely proportional to the rate. These results suggest that the viscosity of the polymer is primarily a function of the bisulphite ion concentration. W.R.M.

Stereoisomerism in Polyesters and Polyamides
Zh. S. Sogomonyants and M. V. Vol'kenshtein

Izvestiya Akad. Nauk S.S.S.R. otdel khim. nauk, (5), 611–620 (May 1957)

Stereoisomerides of high-mol.wt. polyesters and polyamides are made and their relationships to the density and the melting and glass points of the polymers are discussed. G.J.K.

Heterogeneous Chain Polyamides. I— Influence of *N*-Substituents on the Properties of Polyamides from *pp'*-Diaminodiphenylmethane
V. V. Korshak, T. M. Frunze, and E. A. Krasnyanskaya

Izvestiya Akad. Nauk S.S.S.R. otdel khim. nauk, (5), 626–630 (May 1957)

The effect of replacing the amino hydrogen by a methyl or ethyl group upon the softening and melting of amide polymers from bis-*p*-aminophenylmethane and adipic, azelaic, and sebacic acids is studied. The influence of hydrogen bonding of the amide group, and to a lesser degree the interaction of aromatic nuclei, is given as an explanation for the lowering of the softening and melting points with increasing substitution on the nitrogen. G.J.K.

Heterogeneous Chain Polyesters
I— Polyesters from Isomeric Phthalic Acids
V. V. Korshak, S. V. Vinogradova, and V. M. Belyakov

Izvestiya Akad. Nauk S.S.S.R. otdel khim. nauk, (6), 730–736 (June 1957)

Polyesters of terephthalic, *iso*phthalic, and phthalic acids and different glycols of the general formula $HO\cdot[CH_2]_n\cdot OH$ and also diethylene glycol, triethylene glycol, propylene glycol, and 1:3-butanediol are synthesised, and the properties of the polyesters discussed on the basis of the structure of the initial compounds.

II— Polyesters from Isomeric Diphenyldicarboxylic Acids
Idem

Ibid., 737–745

The preparation of *mm'*- and *pp'*-diphenyldicarboxylic acids and their condensation as well as that of diphenic acid with different aliphatic glycols to form polyesters is described.

III— Polyesters from β-Methyladipic and β-*p*-Nitrophenylglutaric Acids
V. V. Korshak and S. V. Vinogradova

Ibid., 746–749

Polyesters of β-methyladipic and β-*p*-nitrophenylglutaric acids with glycols are synthesised and their properties correlated with structural features of the initial substances. G.J.K.

Polymorphism of Bis-β-hydroxyethyl Terephthalates
A. Miyake

Bull. Chem. Soc. Japan, **30**, 361–363 (June 1957)

Four different polymorphic forms (α, β, γ, δ) of bis-β-hydroxyethyl terephthalate have been obtained (α being the normal, most stable modification), and their infrared spectra and X-ray diffraction patterns measured. The terminal hydroxy groups in all forms are hydrogen-bonded; the ester carbonyls in the β and δ forms are free, in the γ

form all are hydrogen-bonded, and in the *a* form free and hydrogen-bonded carbonyls exist in equal amounts. The *β* form has one pair of bands at 909 and 857 cm^{-1}., corresponding to $-CH_2-$ vibration arising from the *trans* configuration of the $-O-CH_2-CH_2-O-$ grouping; the *δ* form has bands at 899 and 870 $cm.^{-1}$ due to the *cis* configuration of the $-O-CH_2-CH_2-O-$ grouping, and the *α* and *γ* forms have both pairs of bands. Four speculative structures are advanced. A.J.

Catalysis of Polyester Formation by Ester Exchange
W. Griehl and G. Schnock
Faserforsch. und Textiltech., **8**, 408–416 (Oct. 1957)
Precondensation in polyethylene terephthalate formation is discussed in terms of the influence of the catalyst, order of reaction, reaction velocity, activation energy, and kinetics. Polyester formation by means of ester exchange is studied, and the course of the reaction followed by viscosity measurements. The reaction is found to be of the first order. W.R.M.

Influence of Low-molecular Compounds on the Photochemical Decomposition of Polystyrene
V. V. Korshak, K. K. Mozgova, and A. P. Zasechkina
J. Gen. Chem. U.S.S.R., **27**, 1866–1873 (July 1957)
In a study of the action of ultraviolet radiation on polystyrene it is shown that low-mol.wt. compounds, e.g. 1:4-diphenyl-1:3-butadiene, reduce the rate of decomposition considerably. G.J.K.

PATENTS

Improving the Printing Properties and Resistance to Boiling Water of Polyethylene
BASF *BP* 784,923
Polyethylene which has been subjected to energy-rich rays to act on it in presence of 1×10^{18}–7×10^{9} mol. per sq.cm. of the polyethylene's surface area of a gas which reacts with it, e.g. O, Cl or F, can be treated in boiling water and be printed well. C.O.C.

Thermoplastic Material Resembling Suède Leather
BF Goodrich Co. *BP* 782,718
Embedding a powdered, non-hygroscopic material in the surface or outer layers of a polymeric composition and then treating with a solvent which dissolves the embedded material but not the composition yields a product which has the appearance and handle of suède leather, high resistance to abrasion, diffuse rejection and is readily cleansed with soap and water. Each constituent of the polymeric composition should contain a single olefinic double bond and the predominant monomer should be a halogenoethylene having 1 or 2 Cl, Br or F atoms on only one C atom. C.O.C.

Complex Aluminium Alkoxides (V p. 101)

Flexible, Vapour-permeable, Water-resistant Thermoplastic Coated Fabric (X p. 108)

Finishing Plastic Sheets or Coatings (X p. 108)

Artificial Deerskin (X p. 108)

XIV— ANALYSIS; TESTING; APPARATUS

Determination of Phenolic Compounds in Water and Effluents
E. F. Mohler and L. N. Jacob
Anal. Chem., **29**, 1369–1374 (Sept. 1957)
In a comparison of the five most widely used methods for the determination of small amounts of phenolic compounds in water, the procedure using 4-aminoantipyrin is shown to be quickest, most precise, and most accurate. L.T.W.

Indigo Method for Evaluating Surface-active Agents
K. Hintzmann and W. Langmann
Melliand Textilber., **38**, 1055–1059 (Sept. 1957)
A method is described for the evaluation of the dispersing and protective colloidal efficiency of surface-active agents. Varying amounts of the substance under test are added to a solution of Indigo in its leuco form. Air is then blown through the system to oxidise the Indigo, a part of which forms a coarse precipitate and the rest remains as a clear blue colloidal solution. The concentration of Indigo remaining in solution is estimated colorimetrically. Other things being equal, the amount of Indigo which remains in solution is proportional to the dispersing power of the surface-active agent. There is good agreement between experimental results and theoretical equations derived from first principles. L.A.T.

Estimation of Sulphite–Cellulose Extract in Syntans
M. P. Balfe
J. Soc. Leather Trades Chem., **41**, 118–119 (April 1957)
The proportion of sulphite–cellulose extract (I) in syntans can be estimated by a methoxyl determination; the method of Clarke (*J. Assocn. Off. Agric. Chem.*, **15**, 136 (1932)) is recommended. It is assumed that the solid matter of I contains one-seventh of its weight of methoxyl. J.W.D.

Colorimetric Estimation of Wool Wax on the Fibre, in Liquors, and in Technical Products
K. Heidler
Chem. Technik, **8**, 160–163 (1956): *Tint. ind.*, (26), 156–165 (April 1957)
The method is based on esterification of the wax with acetic anhydride and sulphuric acid. The reaction with cholesterol (Liebermann) has been modified by reacting in chloroform to give a colour sufficiently stable (5 min.) for estimation in a colorimeter or against coloured solutions. According to the concentration of wax, the resultant coloration varies from emerald to yellow. The gradations are matched against standards prepared by mixing Quinoline Yellow (C.I. Acid Yellow 3) and Alizarine Sapphirol SE (C.I. Acid Blue 43) solutions. S.R.C.

Analytical Aspects of some Organic Compounds. V— 2-Hydroxy-3-naphthoic Acids: Determination of Cobalt
S. K. Datta
J. Indian Chem. Soc., **34**, 238–244 (March 1957)
Thorium and Zr are pptd. almost quant. with 2-hydroxy-3-naphthoic acid and with its nitro, bromo, iodo, acetyl, and nitroso (I) derivatives; I ppts. Ti almost quant. The gravimetric determination of Co and its separation from Ni and other elements with I is described. The Co complex of I is closely similar to that of *α*-nitroso-*β*-naphthol (II), but the reagent itself is more freely sol. in water than is II, and the ppt. is more voluminous. J.W.D.

Microcolorimetric Method for Phthalates
M. H. Swann
Anal. Chem., **29**, 1352–1353 (Sept. 1957)
The condensation of phthalic acid with quinol in presence of conc. H_2SO_4 to form quinizarin, which is yellow in benzene soln. and violet in aq. alkaline soln., is used to detect or estimate phthalates in all types of coatings. Succinates also react to give dihydronaphthazarin, which is red in benzene soln. and blue in aq. alkaline soln. The colours due to succinate are obscured by those formed by phthalates when both are present. L.T.W.

***p*-Nitrophenyldiazoaminoazobenzene (Cadion), a reagent for Cadmium**
V. P. Razumova
J. Gen. Chem. U.S.S.R., **27**, 1433–1438 (June 1957)
Cadion, a very sensitive and specific reagent for Cd^{2+}, is shown to behave as an indicator. In alkaline alcoholic solutions it exists in three different forms— yellow at pH < 11·0, orange-red at pH 11·0–11·65, and violet at pH > 11·65. In agreement with Dwyer (*Austral. Chem. Inst. J. Proc.*, **4**, 26 (1937)) the reaction between Cadion and Cd^{2+} is attributed to adsorption. G.J.K.

Identification of Dyes on Textile Fibres
A. Bode
Melliand Textilber., **38**, 289–296 (March 1957)
A method is described for the identification of dyes on textile fibres, based on the solubility of dyes in various reagents. Results are presented in tabular form. L.A.T.

Use of B.S. 1006 Standards in the Paint Industry
Oil and Colour Chemists' Association, Light Fastness Committee
J. Oil & Col. Chem. Assocn., **40**, 544–568 (July 1957)
A detailed report of a co-operative investigation in two phases. In the first a series of 19 paint panels which had

been exposed alongside a set of blue-wool standards was circulated amongst a number of groups of observers, geographically grouped, and each having a leader responsible for the smooth and correct conduct of the work of assessment. The assessments made by these observers are tabulated and analysed. In the second part of the work each group of observers was responsible for carrying out both the exposure and the assessment, and was provided with blue-wool standards and a set of three test panels, each set being made up from replicates prepared at the same time from red, green, and blue paints. The observers were required to assess the fastness after each of three periods of exposure, and also the fastness of the panel as a whole; a group assessment was then arrived at by discussion amongst the members. These results are also tabulated, plotted, and discussed. It is concluded that there is no serious difficulty in the use of blue-wool standards (B.S. 1006) for the assessment of the light fastness of pigmented systems such as paints. The good measure of agreement obtained in the present work would no doubt improve with increasing experience. J.W.D.

Hiding Power of Pigments and Mixtures of Pigments
S. L. M. Saunders

J. Oil & Col. Chem. Assocn., **40**, 643–660 (Aug. 1957)

Comparisons of the hiding power of pigments must be made at the same concn. in the wet vehicle and not in the dry film. Above a certain pigment content (whether by vol. or by wt.) the contrast ratio (C.R.) of a rutile TiO_2 in different media increased sharply in two cases but decreased in the two others. This may be connected with the state of dispersion of the pigment in the different media. Rutile TiO_2 (I) and a 3:1 mixture of I with blanc fixe (II) show the above decrease in C.R., whereas a 1:1 mixture and mixtures less rich in I do not. With II alone there is a final sharp increase in C.R., but this is eliminated when as little as 5% of I is added. The final decrease in C.R. is shown also by mixtures of I with Sb_2O_3 (III) that contain 50% or more of I; unlike II, III alone shows no final increase in C.R. The behaviour of I alone is shared also by anatase TiO_2, ZnO, and ZnS, but not by Lithopones, Sb_2O_3, and white lead. The difference in hiding power of the two titania increases as the pigment content of both paint and film increases. Pigment hiding power should be quoted together with the pigment–binder ratio at which the measurement was made. The C.R. of four extenders (Paris white, blanc fixe, Dolomite, and Supercel A) shows practically no increase until a comparatively high pigment content is attained (ca. 60% by weight, 25% by vol.) and then increases very sharply. A similar treatment of a number of coloured pigments shows that a plot of C.R. *versus* pigment content on a vol. basis gives a better differentiation amongst the pigments than when a weight basis is used. Paints having 4 different pigment contents in two different media were prepared and admixed with various proportions of rutile TiO_2; C.R. and reflectance measurements were made. The curves obtained (which are reproduced) show that with some pigments hiding power increases as the coloured pigment content increases, up to a point, and then decreases. The optimum varies from additions of ca. 5% with Helio Red to ca. 50% with yellow oxide; it varies also with the total pigment content. Other pigments cause a fairly rapid rise in C.R. with the smaller additions, tailing off as greater additions are made e.g. with vegetable black and red oxide. In these cases, max. C.R. is associated with max. proportion of coloured pigment. J.W.D.

Instrumental Colour Reproduction of Paint
Baltimore Paint and Varnish Production Club Technical Committee on Colour (A. J. Bruning, Chairman)

Off. Dig. Fed. Paint Varn. Prod. Cl., **29**, 1062–1068 (Nov. 1957)

Thirteen colour-measuring instruments representing 9 different types were used to measure a number of selected standard colours in the form of coloured chips and painted panels. The colours ranged from high to low reflectance, from high to low chroma, and included hues distributed around the colour solid. Using measurements only, colours were reproduced to match the standards, the closeness of match to standard being judged by both instrumental and visual means. Metamerism was avoided by using the same ingredients in the standards and the matching samples. Instrumental determinations of the "closeness" of "near-matches" to a standard were also made, using both gloss and flat finishes, and these "near-matches" were then judged visually by men experienced in approving paint matches. A comparison of visual judgments *versus* instrumental results is given. The conclusions reached are—(i) Colour-measuring instruments are quite satisfactory for the reproduction of non-metameric colours from measurements taken some time previously. (ii) The instruments used varied only in ease of operation and interpretation of results; there was no significant difference in precision amongst them. (iii) In selecting the best match from a set of chips the instruments give excellent results in the range 1 NBS unit. The precision of the instruments is of the same order as that of good visual judgment. (iv) In tinting to match a standard, the limiting factor is the patience and experience of the operator rather than the precision of the instrument used. (v) The ability of instruments to match standards that have been lost or have changed, is about equal to that of a trained colour-matcher to match them by direct comparison. J.W.D.

Interference Microscope in Fibre Research. I—Initial Studies on Rayon, Cotton, Wool, and Synthetic Fibres
A. N. J. Heyn

Text. Research J., **27**, 449–458 (June 1957)

The application of the Baker interference microscope in fibre research is described. It is found possible to reveal and distinguish fine details of the fibre structure and to recognise even minute differences in refractive index by colour contrast. It is possible to determine quantitatively the refractive index of these details. The theoretical background is considered and the technique employed described. Typical examples with different fibres are shown. S.B.D.

Attempted Location of the Substituent in Cellulose Xanthate
A. K. Sanyal, E. L. Falconer, D. L. Vincent, and C. B. Purves

Canadian J. Chem., **35**, 1164–1173 (Oct. 1957)

Cellulose sodium xanthate was methylated with methyl iodide in benzene or ether to the *S*-methyl xanthate. This was partially hydrolysed, and the hydrolysate chromatographed on paper. About 20% of glucose was recovered together with two substances recognised as characteristic hydrolytic products of glucose 6-*S*-methyl xanthate. The procedure was not quantitative for xanthate groups in the primary alcohol positions of cellulose, and too drastic to yield information regarding less stable groups in secondary positions. Acetylation of the *S*-methyl xanthate gave a fully acetylated product with unaltered xanthate substitution. Attempts to remove the *S*-methyl xanthate groups resulted in simultaneous removal of acetyl groups. W.R.M.

Gravimetric Estimation of Medulla in Wool
D. A. Ross and J. B. Speakman

Text. Research J., **27**, 345–351 (May 1957)

The residues are weighed after samples of known dry weight have been treated with a 1·6% soln. of peracetic acid at 25°C. for 48 hr., followed by N. ammonia at 25°C. for 24 hr. A correction must be made for the amount of residue given by non-medullated wools, which is approximately the same for Merino and crossbred wools. Samples for analysis should not be dried at high temp. before use, as the amount of residue increases with increasing temperature, especially when the moisture content of the wool is high, owing to the formation of resistant cross-linkages between the peptide chains. S.B.D.

Determination of Hydrogen Peroxide in Bleach Baths
AATCC Committee on Bleaching

Amer. Dyestuff Rep., **46**, P 397 (3 June 1957)

Procedure—to approx. 20 ml. of distilled water add 10 ml. of dil. H_2SO_4. Titrate with 0·588 N-$KMnO_4$ to a point where one drop will give the solution a faint pink colour persisting for 30 sec. The number of ml. required, with the decimal point moved one place to the left, will be the % strength of the bath in terms of 100% H_2O_2. P.T.S.

Standard Depths of Colour in relation to the Assessment of the Colour Fastness of Dyes on Textiles
P. Rabe
Amer. Dyestuff Rep., **46**, P 504–P 508 (15 July 1957)
The results of colour-fastness tests depend on the depth of the dye, which is defined by internationally agreed standards and estimated visually. Uncertainties are caused by differences in hue, brightness, and the nature of the substrate (matt or lustrous). These errors can be eliminated by using an integrating spectrometer and a standard source of light corresponding to natural daylight from an overcast sky. The use of this instrument is described. The magnitude of errors arising from visual estimation is discussed, and examples of results from colour-fastness tests on dyeings at certain fractions of the standard depth are given. P.T.S.

Universal, Digital Tristimulus Integrator
W. E. White and D. L. MacAdam
J. Opt. Soc. Amer., **47**, 605–611 (July 1957)
Selected ordinates (to 0·1 mμ.) for X, Y, and Z are programmed by motorised punched cellulose acetate tape, synchronised with a G.E. spectrophotometer camshaft. Adjustment factors are eliminated by using different numbers of ordinates, so that electronic summation counters display final tristimulus values. R.B.B.

Simple Logarithmic Ratio-recording Spectrophotometer Attachment
P. Hariharan and M. S. Bhalla
J. Opt. Soc. Amer., **47**, 378–381 (May 1957)
Double-beam, single-detector attachment with logarithmic response circuit, to convert any good monochromator into a spectrophotometer recording directly in optical density. The method renders unnecessary servomechanisms or ratio-recording potentiometers and compensates for changes in incident energy and in photocathode sensitivity. Optical densities as high as 3 are measured to better than 0·02 density unit. R.B.B.

New Chromaticity Diagram
R. A. Houstoun
J. Opt. Soc. Amer., **47**, 670–671 (July 1957)
Some speculations on the implications of the asymmetry of the C.I.E. specification (trichromatic coefficients plus luminosity) compared with the symmetry of the tristimulus Young–Helmholtz theory. Mass, momentum, and energy produced by the stimulus replace the three receptor systems. R.B.B.

Combination of Photoreceptor Responses in the Photopic Luminosity Characteristic
C. E. M. Hansel
Nature, **179**, 1290–1291 (22 June 1957)
A brief analysis of the above problem, with special reference to the possible non-linear nature of the summation of photoreceptor response on the trichromatic theory. R.B.B.

Correlate for Lightness in Terms of C.I.E. Tristimulus Values— I
C. L. Sanders and G. Wyszecki
J. Opt. Soc. Amer., **47**, 398–404 (May 1957)
37 Munsell papers of value 5/ are visually evaluated for lightness, using a Munsell neutral scale and 5′, 2°, and 10° fields. Results confirm that C.I.E. calculated luminous reflectances do not correlate with visual sensation except in the yellow region. A second-degree equation in X, Y, Z gives a good approximation. There is some evidence that the violet receptor contribution to light sensation decreases with field size. R.B.B.

Empirical Derivation of the C.I.E. Luminosity Curve from Colour-mixture Data
R. E. Wienke
J. Opt. Soc. Amer., **47**, 622–625 (July 1957)
Scalar additivity of luminance (Abney's law) appears to be invalid, since a good approximation to the C.I.E. luminosity curve could not be generated from radiances weighted with appropriate luminosity coefficients. Incorrect luminosities do not appear to account for the failure, which is apparently due to the non-linear nature of luminance. Possibly vector addition would be more appropriate, though it is doubtful whether it would be worth the additional effort in ordinary colorimetric work. R.B.B.

Chromatic Significance of Spectrophotometric Errors
D. L. MacAdam
J. Opt. Soc. Amer., **47**, 766 (Aug. 1957)
Assumptions made in an article under this title (G. T. Shipley and G. S. Walker, *ibid.*, **46**, 1052 (1956)) represent an unlikely and, at certain wavelengths, an impossible distribution of spectrophotometric errors. Error effects with a properly built and maintained spectrophotometer would not be more than one-third as great as estimated by these authors. R.B.B.

Propagation of Errors in Tristimulus Colorimetry
I. Nimeroff
J. Opt. Soc. Amer., **47**, 697–702 (Aug. 1957)
The theory of propagation of errors in a computed result is applied to chromaticity calculations. Uncertainties in chromaticity co-ordinates for spectrum colours are considered. The importance of specifying variances, along with mean tristimulus values, for any international standard-observer system, is pointed out. R.B.B.

Comparisons among Spectral Sensitivity Data obtained in Different Retinal Locations and with Two Sizes of Foveal Stimulus
H. G. Sperling and Y. Hsia
J. Opt. Soc. Amer., **47**, 707–713 (Aug. 1957)
Relative cone-sensitivity measurements seem to confirm humps in the blue region of the sensitivity function as due to cone activity rather than to macular pigment absorption. Large variations in the relative sensitivity in the blue occur with variation in retinal location and with field size in the fovea. R.B.B.

Horticultural Colour Chart Names with Munsell Key
D. Nickerson
J. Opt. Soc. Amer., **47**, 619–621 (July 1957)
Munsell key for the 800 samples in the two-volume 1939 edition of the *Wilson Horticultural Colour Chart* issued by the Royal Horticultural Society in co-operation with the British Colour Council. R.B.B.

Laboratory Apparatus for Chemical Modification of Cotton Yarn
N. A. Cashen, E. M. Burns, and A. M. DuPré
Text. Research J., **27**, 390–392 (May 1957)
An apparatus embodying three stages separated by molten-metal seals, over which very close control of time and temperature can be made, is described. It may be used to study such reactions as the partial acetylation or the cyanoethylation of cotton yarns in either batchwise or continuous processing. S.B.D.

Rapid Test for Resin Identification on Textiles
J. H. Howard
Amer. Dyestuff Rep., **46**, 313 (6 May 1957)
When acid hydrolysis of the resin is followed by treatment with NaOH, liberation of ammonia indicates a urea–formaldehyde resin. When acid hydrolysis is followed by treatment with phenylhydrazine and ferric chloride the colour complex formed is characteristic of the resin. Ethyleneurea–formaldehyde, melamine–formaldehyde, and epoxy resins can be identified. Acrylonitrile polymer also can be recognised, giving a similar reaction to that of epoxy resin. P.T.S.

Chromatographic Identification of Amino Aldehyde (Resin) Finishes on Textiles
B. Norwick and E. Bierig
Amer. Dyestuff Rep., **46**, 314–315 (6 May 1957)
Descending paper chromatography detects most mixtures of amino aldehyde resin finishes, warns of new and different finishes, indicates the presence of more than urea and melamine, and is independent of the type of fibre, dyeing, or cure. Only extremely small quantities are required for the test. A purified textile sample is hydrolysed in 20 times its weight of 0·1 N-HCl at the boil for 15 min. The hydrolysate is spotted on to filter paper and eluted downwards with an *n*-butanol–ethanol–water

(3 : 1 : 1) solvent. Known and unknown hydrolysates can be run at the same time. The spots are detected by treatment with formaldehyde, followed by curing and staining with Scarlet 4RA (C.I. Acid Red 18), R_f values for urea, melamine, thiourea, dicyandiamine, and cyclic ethyleneurea are given. P.T.S.

Relation of Intrinsic Viscosity to Cellulose Chain Length
W. J. Alexander, O. Goldschmid, and R. L. Mitchell
Ind. Eng. Chem., **49**, 1303–1306 (Aug. 1957)

As the absolute measurement of D.P. is difficult, it is customary to measure the intrinsic viscosity of cellulose or of one of its derivatives, dispersed in a suitable solvent, and calculate the D.P. from this. The intrinsic viscosities of a range of celluloses were determined as cellulose in cupriethylenediamine, cellulose nitrate in ethyl acetate, secondary cellulose acetate in acetone and in cupriethylenediamine (which saponifies the acetate without degradation) and as cellulose xanthate in NaOH. The factor for converting intrinsic viscosity to D.P. differs for each solvent, and except in the case of cellulose nitrate–ethylacetate the factor is not constant over the D.P. range 10–400. A table gives the factors for converting intrinsic viscosity in the above solvents to D.P. at various D.P. levels. W.K.R.

Structure of Cellulose by Alcoholysis
I. I. Korol'kov, V. I. Sharkov, and E. N. Garmanova
J. Appl. Chem. U.S.S.R., **30**, 586–598 (April 1957)

An investigation of the reactivity of ground cellulose during alcoholysis in methyl, ethyl, propyl, and butyl alcohols showed that there is practically no recrystallisation of amorphous cellulose. Ethanolysis can be successfully employed for the determination of the crystallinity of cellulose. By raising the temp. it is possible to decrease the influence of recrystallisation on the rate of hydrolysis of amorphous cellulose and on the yield of sugar formed simultaneously. Hydrolysis of cellulose containing a large proportion of amorphous cellulose is accompanied by rapid recrystallisation. Values for the amorphous fraction obtained from density determinations agreed with the results obtained by ethanolysis. T.Z.W.

Infrared Spectrophotometric Procedure for Analysis of Cellulose and Modified Cellulose
R. T. O'Connor, E. F. DuPré, and E. R. McCall
Anal. Chem., **29**, 998–1005 (July 1957)

The spectrophotometric determination of the infrared absorption spectrum of a compressed disc of intimately mixed ground cellulose or cellulose derivative and potassium bromide provides a simple and rapid method of analysis of any modified or unmodified cellulose fibre, yarn, or fabric for functional groups. Absorption spectra of several cellulose derivatives are given and characteristic bands are listed. L.T.W.

False Viscosity in Cellulose Acetate Solutions
G. A. Richter and L. E. Herdle
Ind. Eng. Chem., **49**, 1451–1452 (Sept. 1957)

For cellulose acetate prepared from acetylation-grade linters, the viscosity of concentrated solutions in acetone can be predicted from the intrinsic viscosity in dilute solutions. For acetates from wood pulps, however, the viscosities of concentrated solutions are higher than predicted and also vary according to the processing history of the wood cellulose. This difference between the viscosity predicted and that found is called the *false viscosity*. A method of measuring false viscosity is described. For acetates from a given cellulose, a straight line is obtained by plotting the logarithm of the viscosity in concentrated solution in acetone against intrinsic viscosity. The ratio of the slope of this line to that given by acetates from cotton linters, less $1{\cdot}00 \times 100$, is the "false viscosity number", which is a characteristic of the cellulose. Wood pulps with a false viscosity number > 15 are unsuitable for commercial acetylation. W.K.R.

Characterisation of Cellulose Derivatives by means of the X-Ray Diffractometer
L. Segal and C. M. Conrad
Amer. Dyestuff Rep., **46**, 637–642 (26 Aug. 1957)

When an efficient Geiger counter is used to measure the intensity of the diffracted beam from an X-ray diffractometer, the accuracy attainable is much higher than with the old, photographic methods. Such an instrument can be used to detect accurately physical and chemical modification of cellulose by various treatments. The change from cotton cellulose I to cotton cellulose III in ethylamine can be followed, and also its reversal in water. The transformation of cotton cellulose I to cotton cellulose II during mercerisation is followed. The method can be used to investigate the course of cyanoethylation of cotton. The effect of hydrochloric acid on the cellulose modifications can be observed. The method gives information about crystallite orientation in various cottons. P.T.S.

Colorimetric Determination of Glycollate in Carboxymethylcellulose
M. Eastwood
Anal. Chem., **29**, 981–982 (June 1957)

Glycollate is washed from sodium carboxymethylcellulose with hot 80% ethanol and determined colorimetrically in a small aliquot of the wash liquor with chromotropic acid in conc. H_2SO_4. L.T.W.

Colorimetric Determination of Tyrosine
G. Ceriotti and L. Spandrio
Biochem. J., **66**, 607–610 (Aug. 1957)

An improved method for the quantitative determination of tyrosine by reaction with α-nitroso-β-naphthol is described. Optimum experimental conditions were selected. The solution of α-nitroso-β-naphthol was made in aqueous 0·1N-NaOH, the exclusion of ethanol from the reaction allowed stabilisation of the colour. Linearity of colour response was obtained between 1 and 16 μg. of tyrosine per ml. The method may be used to estimate the tyrosine content of proteins. P.G.M.

Determination of Tyrosine and Tryptophan in Proteins
W. L. Bencze and K. Schmid
Anal. Chem., **29**, 1193–1196 (Aug. 1957)

In a new spectrophotometric method for the determination of tyrosine and tryptophan in proteins the absorption curve is plotted between 278 and 293 mμ. The gradient of the tangent to the two characteristic maxima of the curve indicates the molar ratio of tyrosine to tryptophan, and the extinction at the higher maximum is a measure of the total tyrosine + tryptophan content. L.T.W.

Identification of Regenerated Protein Fibres by Paper Chromatography
J. Derminot
Bull. Inst. Text. France (65), 7–14 (Feb. 1957)

It is shown that regenerated protein fibres derived from casein (Fibrolane, Merinova), ardein (Ardil), and zein (Vicara) may be distinguished by visual inspection of two-dimensional paper chromatograms of their hydrolysates, after development with ninhydrin. J.C.F.

Simple Device for forming Solution Concentration-gradients
D. B. Wetlaufer
Nature, **180**, 1122–1123 (23 Nov. 1957)

Standard Depths for the Assessment of the Colour Fastness of Dyed and Printed Textiles
P. Rabe and O. Koch
Melliand Textilber., **38**, 173–177 (Feb. 1957)

The results of colour fastness tests depend on the depth of dyeing of the pattern. For this reason it is usual to perform fastness tests on samples dyed to standard depths. Such dyeings can be prepared with the aid of a formula developed for calculating the depth of colour based on saturation, hue, and lightness according to the system of the DIN colour chart. L.A.T.

Suntest R–1041 Fading Lamp
Melliand Textilber., **38**, 188–189 (Feb. 1957)

The Suntest R–1041 fading lamp is based on the American Fade-Ometer, and good correlation exists between the two instruments in the assessment of the light fastness of dyed textiles. Light from a carbon arc surrounded by a partial vacuum is screened by a special Pyrex glass filter. The spectral characteristics of the

transmitted light are said to be similar to those of daylight. There is provision for control of temperature or humidity. Up to 50 test patterns can be accommodated on a revolving sample-holder at 20 cm. from the light source.

L.A.T.

Selection of the Optimum Source of Artificial Illumination for the Matching of Colours
Philadelphia Paint and Varnish Production Club, Illumination Committee (P. Wetherill, Chairman)
Off. Dig. Fed. Paint Varn. Prod. Cl., **29**, 1153–1158 (Nov. 1957)

The investigation sought to establish the best artificial source of illuminations (i) for the matching of colours under plant production conditions, and (ii) for the matching of colours under all conditions, including consideration of metameric matches. Seven light-sources, representing a wide range of types, were used. It was concluded that—(i) for production control within practical limits of illumination intensity and reasonable dispersion of light intensity of the spectrum range, any light-source can be used successfully to match colour, as long as the pigments present are similar in composition; and (ii) multiple light-sources are required to establish an all-purpose viewing condition for the matching of colours involving metameric pigments. Two light-sources of different colour temp. are involved, and colours should be matched under one condition (7500°K.) and then verified at another (2800°K.), or similar contrasting conditions are necessary. It is recommended also that colour matching should be conducted in a box having interior dimensions of 3 ft. × 3 ft. × 2 ft. deep and painted a neutral grey. Eye fatigue is promoted if the illumination intensity exceeds 150 ft.candles. A diffusing glass should be used to secure even light distribution.

J.W.D.

Tolerances in Colour Matching
D. L. Tilleard
J. Oil & Col. Chem. Assocn., **40**, 952–975 (Nov. 1957)

Measurements have been made with the Beckman photoelectric spectrophotometer, with reflectance attachment, and the results compared with judgments made by colour matchers. Analysis of the measurements so made indicates that the precision to be expected from a single measurement with this instrument on a fairly light colour is about 0·3 N.B.S. unit. Increased precision may be obtained by averaging several measurements. For dark or highly saturated colours the precision is much lower. With an instrument of this type, there is evidence that decorative finishes, at least those of a light colour, can be measured and compared within a commercially significant tolerance. Many stoving finishes require a precision of measurement of 0·4 N.B.S. unit, or better, but the instrument can give useful results in such close matching if used at its highest precision. The discrimination of the instrument would not be sufficient to distinguish the smallest differences perceptible under the best conditions, except possibly for near-whites.

J.W.D.

Colour Vision of Achromats' Parents
R. W. Pickford
Nature, **180**, 926–927 (2 Nov. 1957)

Determination of Melamine-Formaldehyde Resins in Coatings
M. H. Swann and G. G. Esposito
Anal. Chem., **29**, 1361–1362 (Sept. 1957)

The insolubility of acid-hydrolysed melamine resins in dioxan provides a rapid gravimetric method for their determination in modified alkyd, epoxy, and other coating resins.

L.T.W.

"Labelling" of Synthetic Resins in Resin-treated Fabrics with Fluorescent Dyes
H. Ruile
Melliand Textilber., **38**, 442–448 (April 1957)

After critical discussion of the existing staining methods the author describes the application of fluorescent dyes to the study of the uniformity of distribution and penetration of resins in resin-treated fabrics. A fluorescent dye is added to the resin precondensate bath, or it can be applied to the finished fabric. The material is then examined visually under ultraviolet radiation or with the aid of a microscope adapted for fluorescence work.

L.A.T.

Radioactive Isotopes for the Study of the Distribution of Resins in Resin-treated Fabrics
H. Enders
Melliand Textilber., **38**, 547–555 (May 1957)

Besides describing existing methods for the study of the distribution of resins in resin-treated fabrics the author gives an account of his technique based on the use of radioactive isotopes. In the experimental work described resins containing ^{14}C were employed. Fabric or fibre cross-sections were brought in contact with a photographic plate. From the autoradiograph thus obtained the exact location and distribution of the resin can be deduced. The advantages and disadvantages of the method are discussed.

L.A.T.

Proposed Official Method for Fastness of Coloured Leather to Water
Society of Leather Trades Chemists, Fastness Tests Committee (J. C. Pittard, Chairman)
J. Soc. Leather Trades Chem., **41**, 125–126 (April 1957)

A specimen of the finished leather in contact with the relevant uncoloured material is immersed in water and covered with a piece of glass. After 15 min. the water is poured off without removing the plate. Having been kept for 4 hr. at 37 ± 2°C. the specimen and the uncoloured material are dried separately. The change in colour of the specimen and the staining of the uncoloured material are assessed with the Geometric Grey Scale.

J.W.D.

Proposed Official Method for Testing the Fastness to Rubbing (Wet and Dry) of Light Leathers
Society of Leather Trades Chemists, Fastness Tests Committee (J. C. Pittard, Chairman)
J. Soc. Leather Trades Chem., **41**, 120–125 (April 1957)

The test-piece is clamped on a horizontal platform and a felt pad 1 in. in diameter is revolved at 150 ± 5 r.p.m. against its surface under a total weight of either 1·6 lb. or 5·5 lb. Test runs of 64, 128, 256, 512, 1024, and 2048 revolutions are made in different locations. The changes in appearance are described in terms such as weaker, darker, greener, after the sample has been wax-polished and examined under standard illumination. The Geometric Grey Scale is used in assessing the results. The methods are described fully and there are diagrams of the apparatus.

J.W.D.

Proposed Official Method for Fastness of Coloured Leather to Perspiration
Society of Leather Trades Chemists, Fastness Tests Committee (J. C. Pittard, Chairman)
J. Soc. Leather Trades Chem., **41**, 127–128 (April 1957)

Specimens of the finished leather in contact with the relevant uncoloured material are treated in soln. containing histidine and NaCl and covered with pieces of glass. After 45 min. the soln. are poured off without removing the glass plates. Having been kept for 4 hr. at 37 ± 2°c., the specimens and pieces of uncoloured material are dried separately. The change in colour of each specimen and the staining of the uncoloured material are assessed with the Geometric Grey Scale.

J.W.D.

Leather–Finish Adhesion Test
V. G. Vely, N. D. Gallagher, and M. B. Neher
J. Amer. Leather Chem. Assocn., **52**, 622–633 (Nov. 1957)

Two new heat-set adhesives have been found which are suitable for use with the leather–finish adhesion test; they are applicable to practically all types of leather finish. One is based upon a mixture of polyamide resins, the other upon a vinyl chloride–acetate copolymer. A solvent-free, cold-set adhesive based upon an epoxy resin has also been found; this adheres well at room temp. to most shoe upper finishes, thus minimising temp. effects during testing. The adhesion values obtained with this latter adhesive are more realistic for some leather finishes than those obtained with the heat-set adhesives; the cold-set method is not applicable to leathers finished with lacquers or vinyl resins. The heat-set adhesives are best adapted to control processes since only a short time is required for preparation of the samples; the cold-set method is more valuable as a research tool.

J.W.D.

Measurement of Molecular Orientation in Filaments of Polyethylene Terephthalate using Dichroic Dyes
D. Patterson and I. M. Ward

Trans. Faraday Soc., **53**, 1517–1526 (Nov. 1957)

A method is described for measuring orientation factors using the dichroism of dyes extruded with the molten polymer when filaments are prepared. Orientation factors are compared with those depending on polarisation anisotropy of the polymer molecules themselves as determined by birefringence measurements. Changes occurring when filaments are drawn are interpreted in the light of changes in the two orientation factors. In non-crystalline but oriented filaments the two methods are equivalent, but in crystalline filaments the birefringence method measures orientation of both crystalline and non-crystalline regions, while dichroism measures only that of the non-crystalline. W.R.M.

Measurement of Hydroxyl and Carboxyl End-groups in Polyethylene Terephthalate
I. M. Ward

Trans. Faraday Soc., **53**, 1406–1412 (Nov. 1957)

The total end-group content is obtained by measuring the deuterium content of water from combustion of deuterated polymer. The intensities of the alcoholic and carboxylic OH infrared absorptions are measured. A calibration method is described for determining the number of each kind of end-group. Use of the method in determining the number-average molecular weight $\overline{M}_n$ is discussed, and results are given for samples of differing molecular weight. The results can be described by the equation $[\eta] = 3 \times 10^{-4}\,\overline{M}_n^{0\cdot77}$, where $[\eta]$ is the intrinsic viscosity in chlorophenol. W.R.M.

Fractionation of Polyacrylonitrile
H. Kobayashi

J. Polymer Sci., **26**, 230–233 (Nov. 1957)

Benzene or toluene is added to a solution of the polymer in hydroxyacetonitrile and dispersed in the solution. When ethanol is added to the mixture, the benzene or toluene dissolves in the polymer solution and the polymer precipitates. Molecular weights of fractions are obtained from the equation—

$$[\eta] = 8\cdot96 \times 10^{-4} M^{0\cdot68}$$

where $[\eta]$ is the intrinsic viscosity in dimethylformamide at 35°c. W.R.M.

Flammability of Thin Flexible Materials
C. T. Webster

J. Textile Inst., **48**, T 471–T 473 (Dec. 1957)

In a previous publication (D. I. Lawson, C. T. Webster, and M. J. Gregsten, *ibid.*, **46**, T 453 (1955)) an expression was derived relating the vertical speed of flame propagation V in./sec. to the time of burning on a semicircular arched track T sec.—

$$V = \frac{650}{T^{1\cdot00}} \qquad \text{(i)}$$

If the flame was not propagated completely round the circle, but went out after it had travelled d in., then the expression was—

$$V = 0\cdot71d^{0\cdot4} \qquad \text{(ii)}$$

In the present paper a third expression is derived from results with 29 textile materials containing wool, cotton, Orlon, viscose rayon, nylon, polyacrylonitrile, Fibrolane, and silk—

$$V = \frac{0\cdot31d^{2\cdot5}}{T} \qquad \text{(iii)}$$

A comparison of actual vertical flame speeds and vertical flame speeds calculated from speeds on the semicircular track shows good agreement. P.T.S.

Evaluation of Carrotted Hair by Chemical Tests. II— Results from Practice
H. G. Fröhlich

Z. ges. Textilind., **59**, 202–206 (20 March 1957): *Chem. Abs.*, **51**, 11724 (10 Aug. 1957)

Of a number of tests applied to factory-carrotted rabbit hair, the acid solubility curve gave the best correlation with the felting power of the treated hair and the quality of the resulting felt. The permanganate number is the best indication of the dyeing behaviour of the carrotted hair. C.O.C.

Image Intensities in Electron Micrographs
S. Asunmaa

Svensk Papperstidning, **60**, 708–712 (15 Oct. 1957)

The possibilities of a quantitative interpretation of the image intensity variations in electron micrographs of ultrathin sections of wood cells without electron contrast are discussed on the basis of Uyeda's theory of image formation. A series of electron micrographs of wood illustrate the scattering absorption in large areas and phase contrast in dimensions of the order of the resolution. G.S.B.

Determination of Carboxyl Groups in Cellulose
O. Franzon, O. Samuelson, B. Bodforss, and E. Ehn

Svensk Papperstidning, **60**, 706–707 (15 Oct. 1957)

The removal of acid from cellulose fibres in the washing-out operation has been studied using radioactive hydrochloric acid. The radiation was measured directly on the cellulose specimen, and experiments showed that the hydrochloric acid is easily washed from the fibres. G.S.B.

Rapid Observation of Phase Transformations in Fibrous Proteins
A. R. B. Skertchly

Nature, **180**, 802–803 (19 Oct. 1957)

Apparatus and technique for rapid observation of the X-ray diffraction image during stretching experiments are outlined. W.R.M.

Titration of Mixtures of Acids
D. Sims and L. Peters

Nature, **180**, 805–806 (19 Oct. 1957)

It is usually assumed that, in the titration of weak dibasic acids, if $K_1/K_2 > 4$, where K_1 and K_2 are the two dissociation constants of the acid, the titration curve can be distinguished from that of a monobasic acid. Calculated titration curves show that K_1/K_2 must be much larger than 4 before the end-point of the weaker acid can be detected. It is shown that K_1/K_2 must be $> 59{\cdot}7$ before each group can be distinguished on the titration curve. It follows that for a polymer containing a_1 amide groups and a_2 carboxyl groups the D.P. (a_1/a_2) must not be too large, or the end-point corresponding to complete titration of the carboxyl groups will not be detectable. W.R.M.

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W. Harrison *BP* 787,589

Comparative Study of the Behaviour at High Temperatures of Partially Acetylated Cotton and some other Fibres (VI p. 102)
Amino-acid Sequence in a Fraction of Fibroin (VI p. 102)
Application of the Oil-immersion Method to the Analysis of a Striation Fault in a Wool–Polyester Fibre Fabric (VI p. 102)
Chemistry of Cellulose Degradation (XI p. 109)

XV— MISCELLANEOUS

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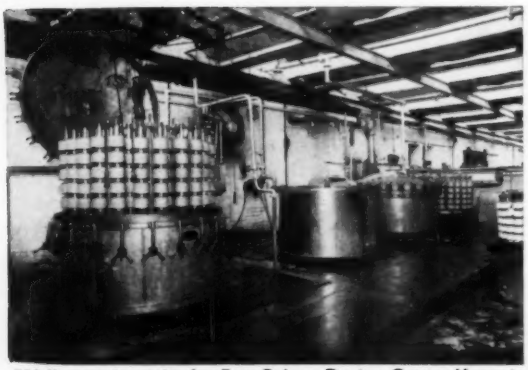

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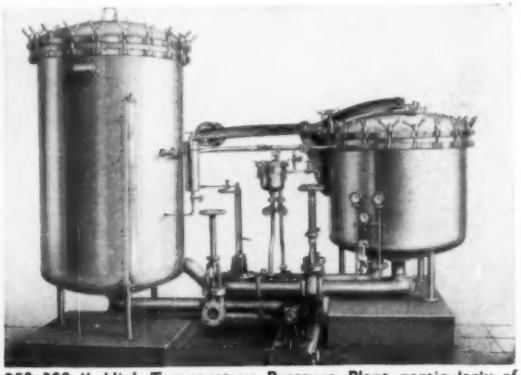

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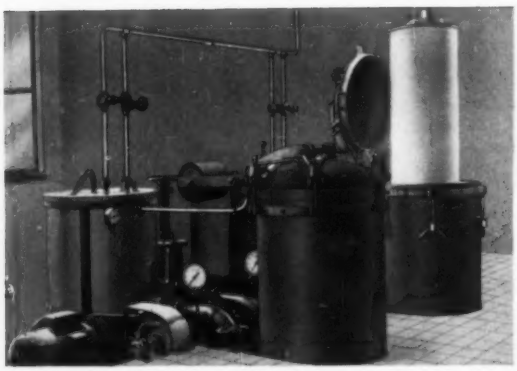

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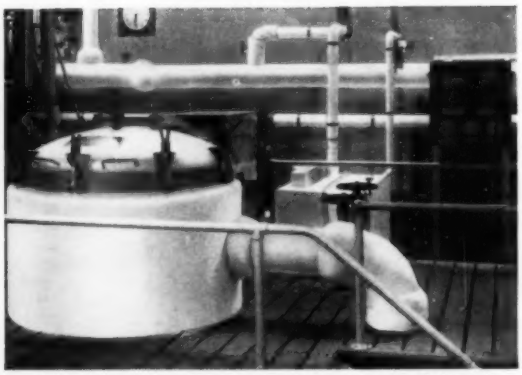

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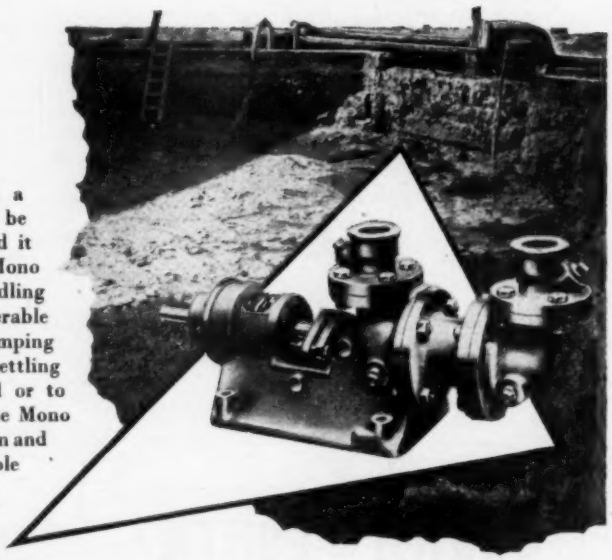

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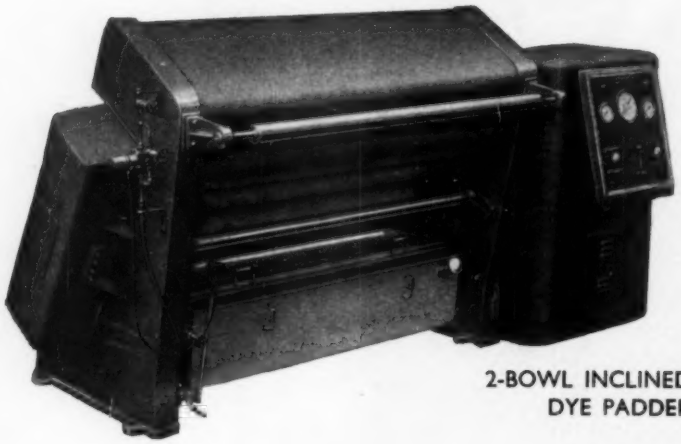

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